# UNIT-I OVERVIEW OF MATERIALS
(Introduction to materials Testing)

## Types of materials:

* Materials can be divided into the following categories
  - crystalline
  - Amorphous

## Crystalline Materials:

* These are materials containing one or many crystals. In each crystal, atoms or ions show a long range periodic arrangement.
* All metals and alloys are crystalline materials.
* These include iron, steel, copper, brass, bronze, aluminium, duralumin, uranium, thorium, etc.

## Amorphous material:-

* Amorphous refers to materials that do not have regular, periodic arrangement of atoms
* Glass is an amorphous material.

Another classification:-

- Metals
- Ceramics
- Polymers
- Composites

Metals:-

* Ferrous (iron & steel)
* Non ferrous metals and alloys

Ceramics:-

* Structural ceramics (High temp. load bearing)
* Refractories (corrosion resistant, insulating)
* Whitewares (Porcelains)
* Glass
* Electrical ceramics (capacitors, insulators, transducers)
* Chemically Bonded Ceramics (Cement and concrete)

Polymers

* Plastics
* Elastomers

Composites:-

* Particulate composites
* Laminate Composites
* Fiber reinforced Composites

# Engineering Materials

- Metals
  - Ferrous
    - Steels
    - Stainless steels
    - Tool and die steels
    - Cast irons
  - Non ferrous
    - Alluminium
    - Copper
    - Titanium
    - Tungsten
    - others
- Plastics
  - Thermo Plastics
    - Acrylics
    - ABS
    - Nylons
    - Polyethylenes
    - PVC
    - others
  - Thermosets
    - Epoxies
    - Phenolics
    - Polyimides
    - others
  - Elastomers
    - Rubbers
    - Silicones
    - Polyurethanes
- Ceramics and others
  - Oxides
  - Nitrides
  - Carbides
  - Glasses
  - Graphite
  - Glass ceramics
  - Diamond
- Composites
  - Reinforced Plastics
  - Metal-Matrix
  - Ceramic-matrix
  - Laminates
  - others

## Properties of materials

* Mechanical properties
* Electrical properties
* Dielectric properties
* Magnetic properties
* Optical properties
* Corrosion properties
* Biological properties

## Mechanical properties

* Elasticity and stiffness
* Ductility
* Strength
* Hardness
* Britteleness
* Toughness
* Fatigue
* Creep

## Electrical properties

* Conductivity
* Resistivity

## Classification of material Testing:

1. Destructive Testing
2. Non Destructive Testing

## Destructive Testing:

* Destructive testing includes methods where your material is broken down in order to determine mechanical properties, such as strength, toughness and hardness.

### Benefits:

* It verifies properties of a material
* It determines quality of welds
* It helps you to reduce failures, accidents and costs.
* It ensures compliance with regulations.

### Types of destructive testing

* compression test
* Tensile test
* Hardness test
  - Rockwell
  - Brinell
  - Vicker's
* Impact test
  - IZOD
  - Charpy

* Compression testing is a very common testing method that is used to establish the compressive force of a material.

* Tensile testing is a destructive test process that provides information about the tensile strength, yield strength, and ductility of the metallic material.
* A hardness test is a method employed to measure the hardness of a material. Hardness refers to a material's resistance to permanent indentation.
* The five most common hardness scales are:
  * Knoop
  * Vickers
  * Rockwell
  * Brinell
  * Shore
* An impact test is a technique for determining the behaviour of material subjected to shock loading in bending, tension and torsion.
* Metal industry sectors that use the impact test is listed below.
* Oil and gas * Aerospace * Power generation
* Automotive * Nuclear

## Non destructive testing (NDT)

* NDT is the process of inspecting, testing or evalluating materials, components or assemblies for discontinuities without destroying the ~~the~~ serviceability of the part or system.

### Benefits:-

* Less waste of samples
* Less downtime
* Accident prevention
* Increased product reliability

### Types of Non destructive testing

* Liqcuid penetrant test
* Magnetic particle test
* Thermography test
* Radiographic
* Eddy current
* Ultrasonic
* Acoustic emission

### Liqcuid penetrant test

* It is easy to use and economical

* The specimen is coated with a red liquid dye which soaks into the surface crack or flaw
* The liquid is then washed off and the part dried
* A developer is sprayed on the part
* Flaws and cracks show up red against the white background of the developer

Magnetic particle test

* This test is used to detect flaws on or near the surface of iron-based metals.
* The part is first magnetized.
* It is then either dusted with fine iron powder or coated with a solution in which iron particles are suspended.
* Flaws in the workpiece cause the lines of magnetic force to become distorted and break through the surface.
* There they attract concentrations of the iron particles, which reveal defects in the metal.

## Purpose of Testing

* To maintain the quality and consistency of the finished product.
* To avoid mistakes in the first stage of the manufacturing process.
* To obtain compliance certification by following guidelines and regulations of testing and by obtaining standard limit of material properties
* To ensure that the materials are suitable for production and usage
* To determine the reason behind product failure during manufacture or while in use.
* To prevent failure in usage.
* To check the components prior to the final assembly.
* To check the component in service without damage
* Used for research and development of existing and new materials.

## selection of material

* material selection is one of the foremost functions of effective engineering design as it determines the reliability of the design in terms of industrial and economical aspects.

## steps for selection of materials

### 1. Identify the design requirements

* The design requirements include the following items
* performance requirements
* Simplicity and practicability
* Reliability requirements
* size, shape and mass requirements
* cost requirements
* Manufacturing and assembly requirements
* Industry standards
* sustainability requirements

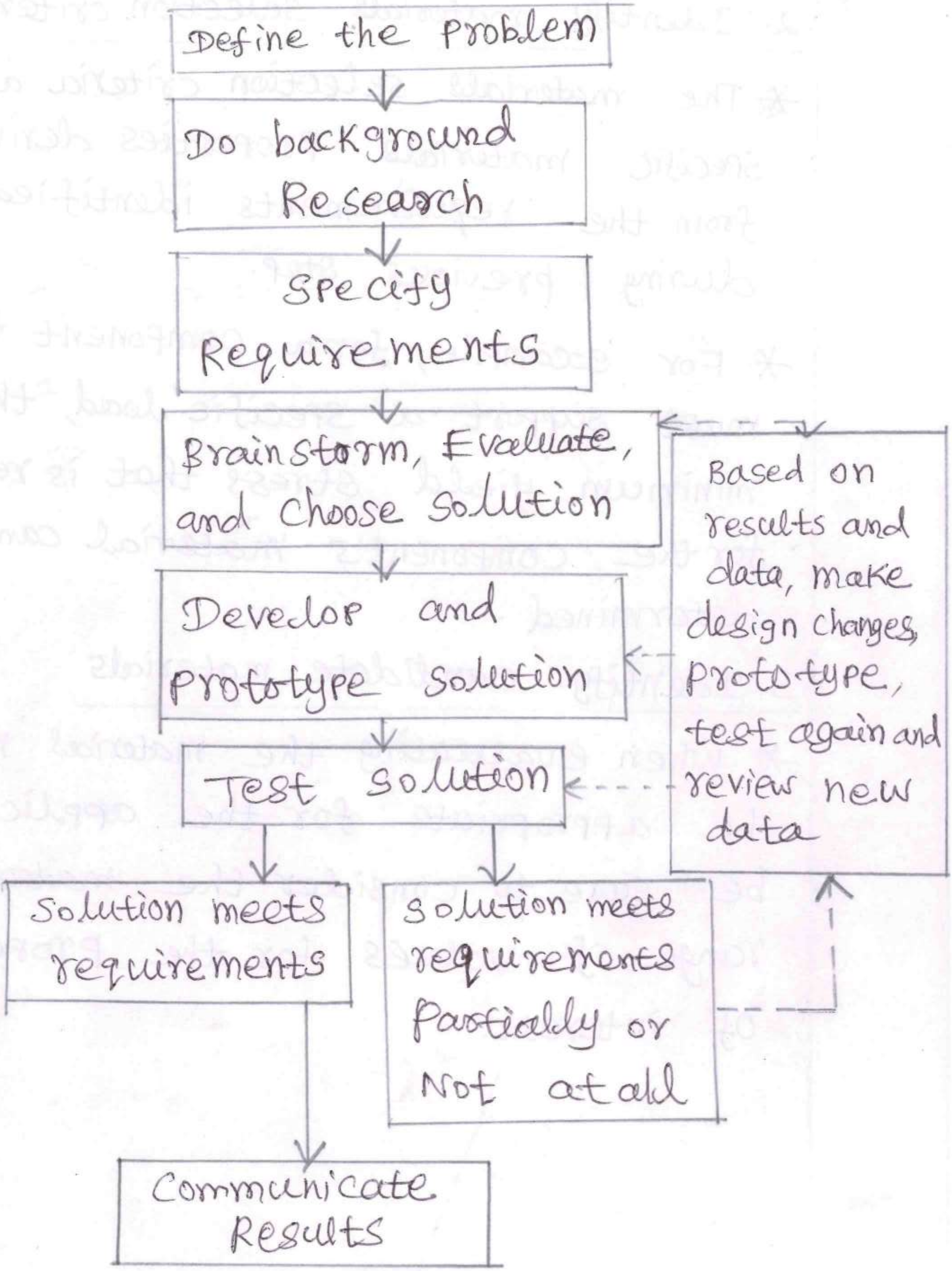
## 2. Identify materials selection criteria

* The materials selection criteria are specific materials properties derived from the requirements identified during previous step.

* For example, for a component that must support a specific load, the minimum yield stress that is required for the component's material can be determined.

## 3. Identify candidate materials

* when evaluating the material might be appropriate for the application, be sure to consider the materials range of values for the properties of interest.




## Failure analysis

## 4. Evaluate Candidate materials

* There may be candidate materials for which there insufficient data available to indicate whether the materials satisfy certain selection criteria.
* These materials to be analyzed and tested to determine whether they meet the selection criteria by creating a prototype.

## 5. Select materials

* select the materials that satisfy all the materials selection criteria at the low cost.
* cost includes the cost of the material and the cost to fabricate a component.

## 6. Failure analysis

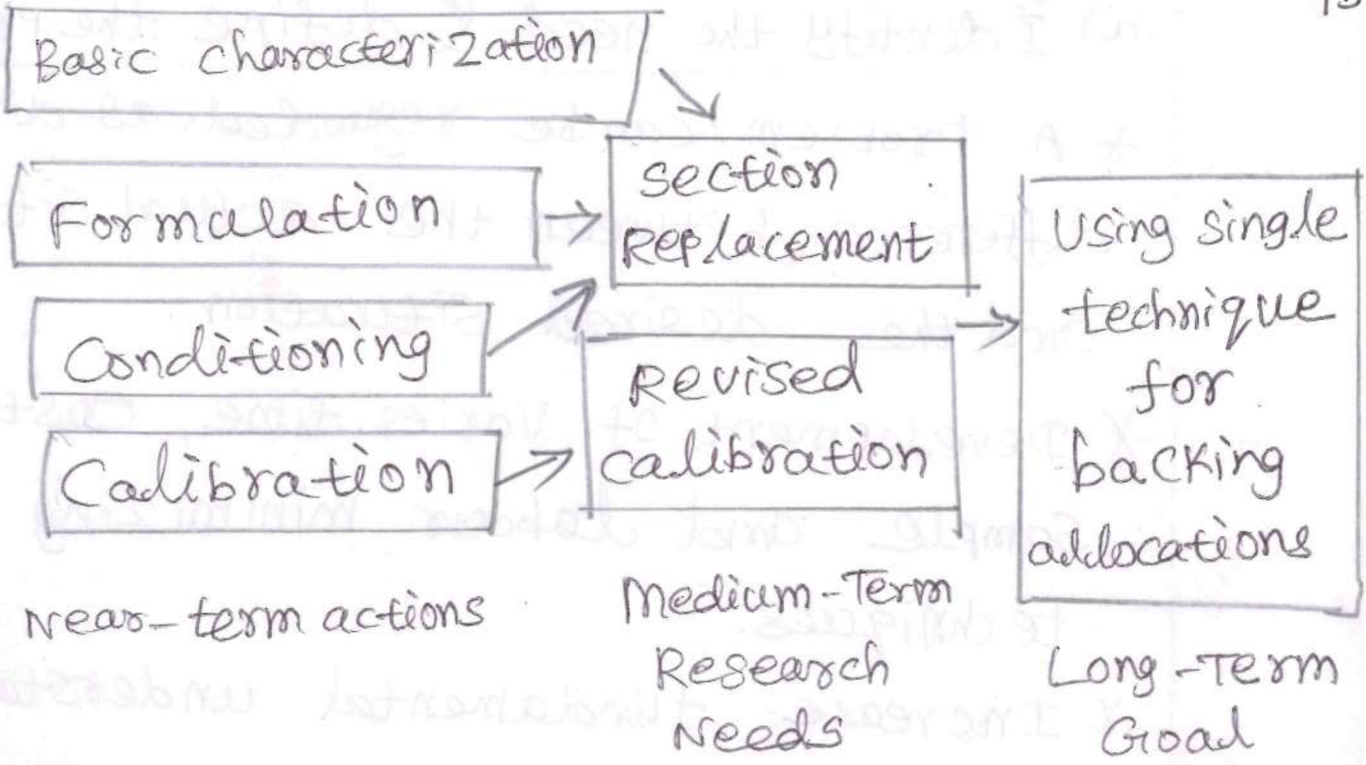
* The Selection of materials is finalized with help of failure analysis mode which is given in the previous figure.

## 7. service experience

* Design changes may also be made as a result of experience with a limited production run of a new product.
* Purchasers of the product may also use it in a way not anticipated by the designer, resulting in failure.
* The design process often continues even after a product is established and widely distributed.

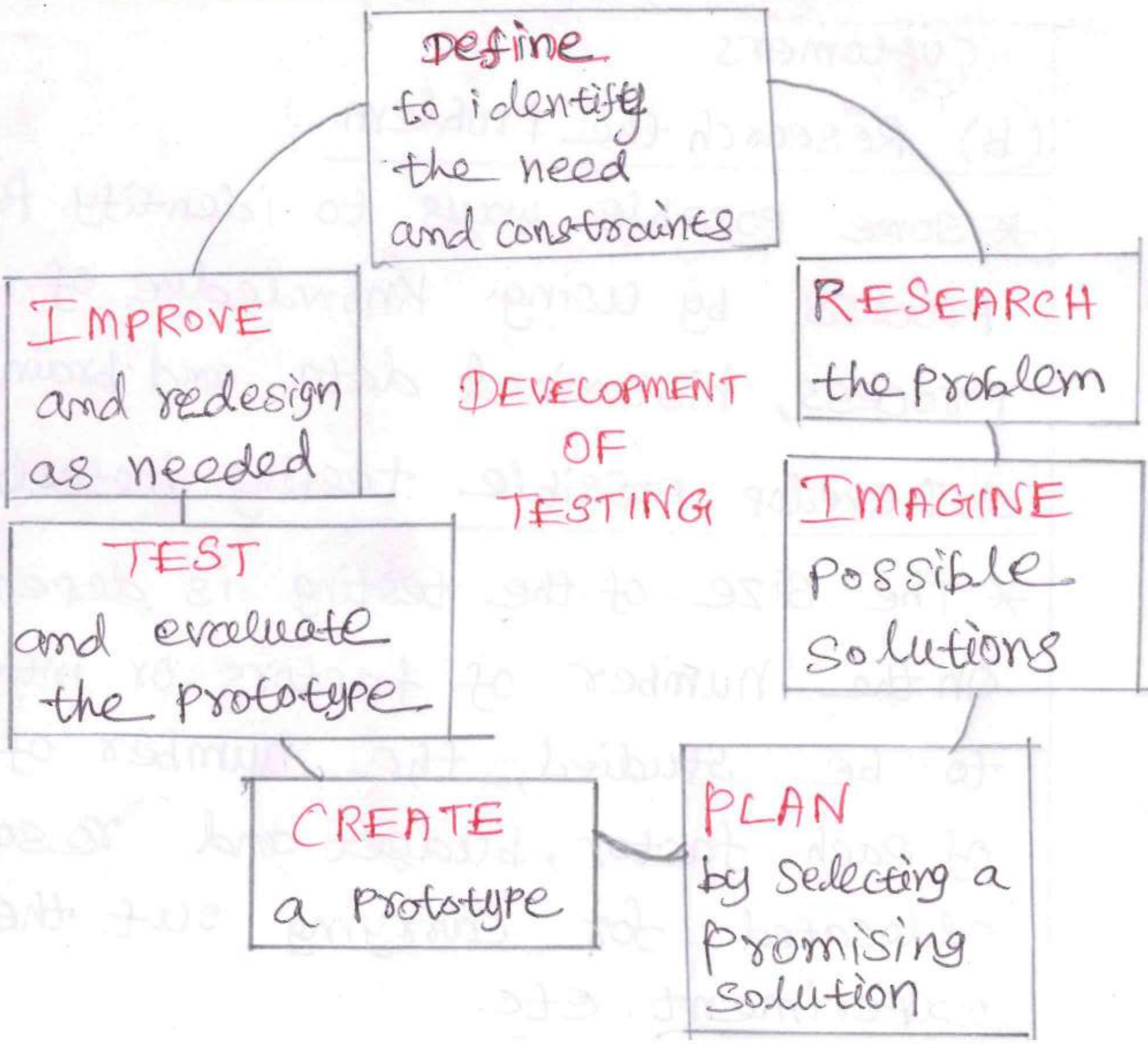
## Development of testing

* measurement of the characteristics and behavior of such substances as metals, ceramics or plastics under various conditions.
* standard test methods have been established by such national and international bodies as the International organization for standardization (ISO).




## Formulation in test development

### Stages in development of testing



(a) Identify the need & define the problem

* A problem can be regarded as a difference between the actual situation and the desired situation.
* Development of varies time, cost, sample and labour minimizing test techniques.
* Increase fundamental understanding of materials
* Improvement of the product performance relative to the needs and demands of customers.

(b) Research the problem

* Some possible ways to identify potential process by using knowledge of the process, historical data and brainstorming.

(c) Develop possible testing methods

* The size of the testing is dependent on the number of factors or interactions to be studied, the number of levels of each factor, budget and resources allocated for carrying out the experiment, etc.

* The development testing plan methods is done using various techniques of graphical presentation such as AutoCAD, CREO and ANSYS, etc.
* The material testing code book gives the basics of testing development standards, which is based on environment, material specification and result analysis methods, etc.

(d) Evaluate the alternatives & select most promising methods

* The various possible method is developed and stimulated in softwares to ensure the theoretical acceptance.
* presenting the information of testing methods are deciding criteria of effective method.
* The testing methods need to satisfy the basic criteria like cost and time.

(e) Initial design

* The initial design is often made on the basis of avoiding stresses that exceed the yield strength of the material.
* Then the design is checked by more refined analysis, and changes are made necessary to avoid modes of material failure.

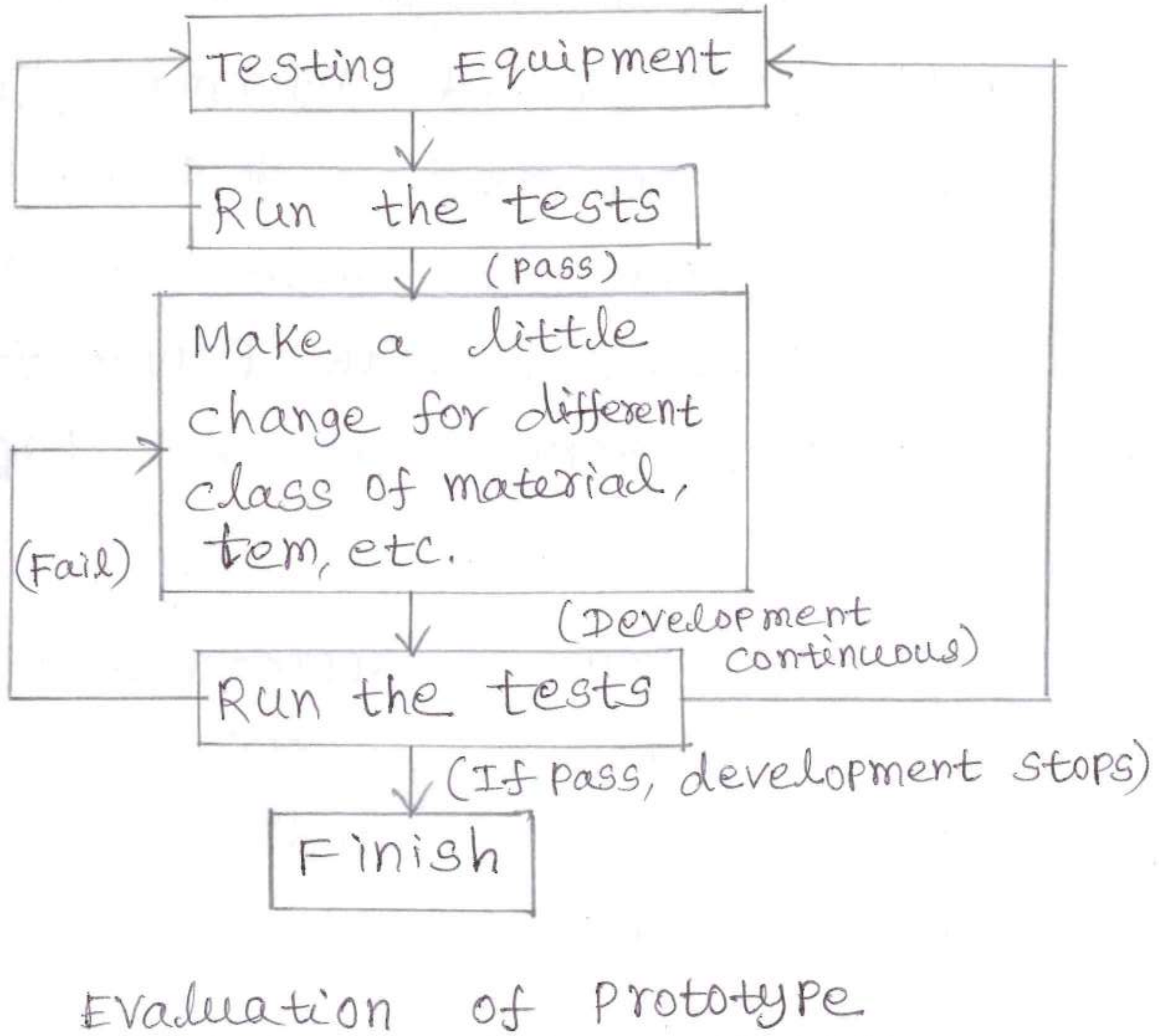
* In making design decisions that involve Safety and durability, the concept of a safety factor is often used.
* The safety factor in stress is the ratio of the stress that causes failure to the stress expected to occur in the actual service of the component.

(f) construct a prototype

* The materials that will be used in final testing methods may be expensive or difficult to fabricate, so prototypes may be made from different materials than the final product.
* A prototype or trial model is often made and subjected to simulated service testing to demonstrate whether it is functions properly.
* Prototypes are generally made with much closer individual inspection and the assumption that some adjustment or rework will be part of the fabrication process.

## (g) Test and Evaluate the prototype

* It is important to test and evaluate the prototype along the way for functionality, usefulness and safety.
* The final product may be subject to a number of quality assurance tests to verify conformance with drawings or specifications.




## Evaluation of Prototype

* The failure of prototype leads to choosing alternatives and redesign the section

* A prototype may also be subjected to simulated service testing until either a mechanical failure occurs, perhaps by fatigue, creep, wear and corrosion. The design is proven to be reliable. This is called durability testing.

(h) Communicate the design

* Communication design is a mixed discipline between design and information development which is concerned with such as printed, crafted, electronic media or presentations to communicate with people for overcoming some unreliable problems.

(i) Redesign

* The redesign is approached existing testing techniques is outdated for the present materials and to minimizing the calibration.

## Testing organizations and its committee

### 1. International organization for Standardization (ISO)

* The international organization for Standardization (ISO) is an international standard setting body composed of representatives from various national standards organizations.
* ISO is a voluntary organization whose members are recognized authorities on standards, each one representing one country.
* Members meet annually at a general assembly to discuss the strategic objectives of ISO. The organization is coordinated by a central secretariat based in Geneva.

### 2. ASTM International

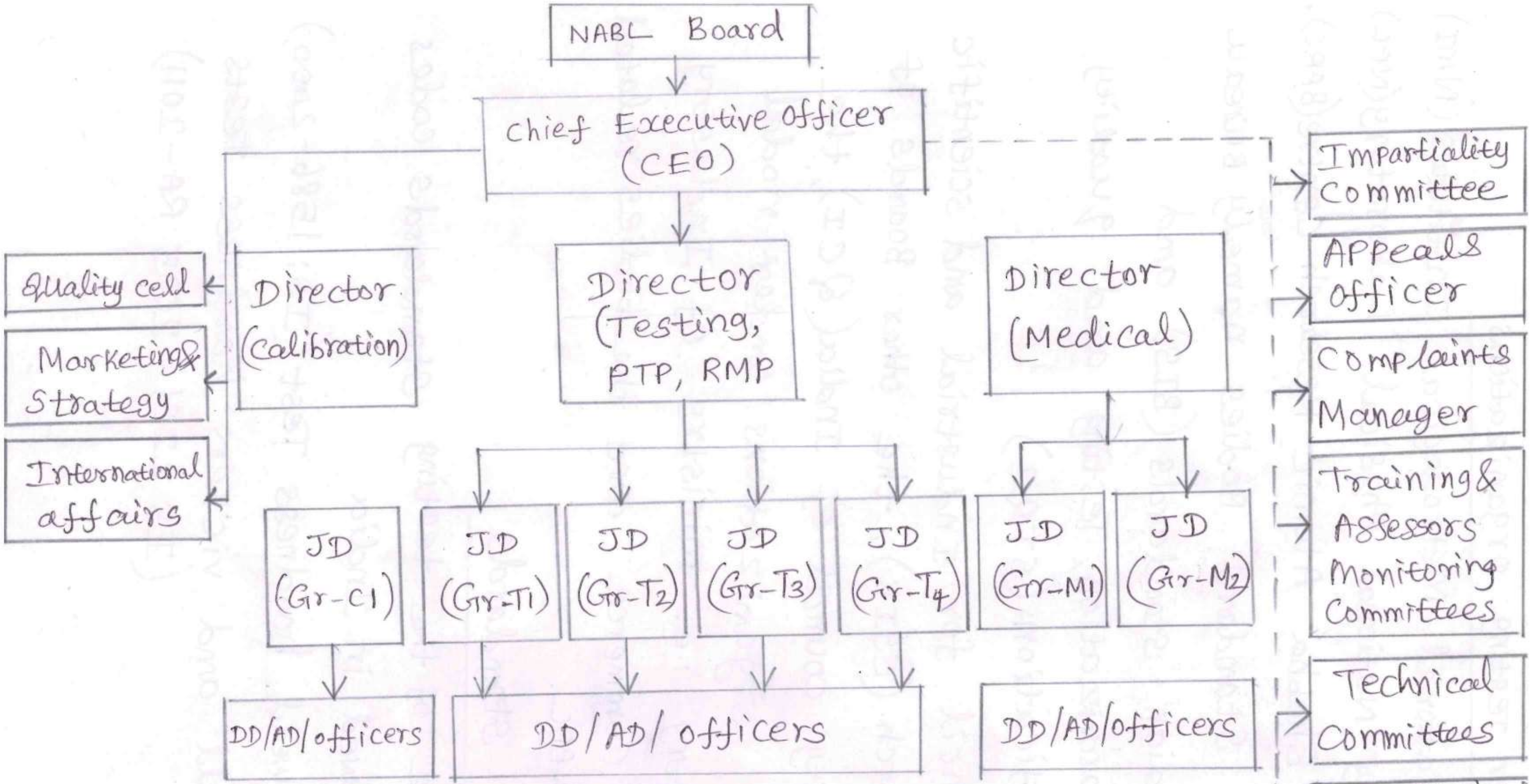
* ASTM international, formerly known as American Society for Testing and Materials, is an international standards organization that develops and publishes voluntary consensus technical standards for a wide range of materials, products, systems and services.

## 3. Bureau of Indian Standards (BIS)

* BIS is the National Standard Body of India established under the BIS Act 2016 for the development of the activities of Standardization, marking and quality certification of goods and for matters connected therewith.
* BIS has been providing tangibility benefits to the national economy in a number of ways
* Providing Safe reliable quality goods
* Minimizing health hazards to consumers
* Promoting exports and imports substitute

### (i) organization of BIS

* The organization of BIS consists of following members
* Governing council Members
* Executive committee
* Administrative Structure




NABL – National Accrediation Board for Testing and Calibration Laboratories

JD – Junior Director

PTP – Proficiency testing Providers

RMP – Reference material Producers

organization chart

## (iii) other Testing organizations

* The National Metrological Institutes (NMI) namely National Physical Laboratory (NPL) and Bhabha Atomic Research Centre (BARC).
* The Standard Bodies namely Bureau of Indian standards (BIS) and Standardization, Testing and quality certification (STQC)
* council for Industrial and scientific Research (CSIR), the other Boards of Quality council of India (QCI), the other organizations under nodal ministry i.e. ministry of Industry and commerce are the bodies related to NABC.

## Testing Standards

* some of the testing standards codes followed in India

1. Rockwell hardness Test (IS: 1586-2000)
2. Brinell and vickers hardness Tests (IS 2281 - 2005 RA-2011)

3. Impact tests (Charpy V-Notch and IZOD Tests) (IS:1757-1988 and IS:1598-1977 RA-2009)

4. Tensile Test (IS:1608-2005 RA-2011)

5. Compression Test (IS:1608-2005/ISO 4506-1979)

6. Bend Test for metal products (IS: 1599-1985 RA-2011)

7. Shear Test (IS:5242-1979 RA-2006)

8. Beam or flexural bending Test (IS: 16-1959)

9. Torsion test and Fatigue test (IS:5074-1969 RA-2001 and IS:5075-1985 RA-2001)

10. Indian Standard Mechanical Testing of Metals Tensile Testing (IS:1608-1995)

11. Metallic materials - Bend Test (IS 1599-2012)

* For all the tests described in this section, the method as specified in relevant ISO Standard may also be followed as an alternate method.

Advantages of Testing.

1. Safety issues can be identified

* The tests are carried out to ensure product safety, and also to make sure the person carrying out the work on any machinery or components is safe too.

* In mechanical testing, the equipment testing area is covered with glass plate to present from shattering of test piece out of equipment.

* most non-destructive tests are harmless to humans, although tests involving radiographic must be carried out under strict settings. All tests must ensure that products are left completely undamaged.

2. It provides reliability

* If workers in industry want reliable and accurate results, all material testing will offer stability.

3. It is cost effective

* These types of tests can also insights that can result in the effective replacement or repair of components or equipment before a real malfunction or breakdown occurs, which will save more money in the long term.

4. It offers reassurance

* Reassurance is a simple thing, but it can sometimes be the most important advantage to testing methods.
* The operation of testing equipment being harmless and it also help to prevent injury.
* When workers know they are safe, they feel more secure and this is something that can benefit productivity, and output.

## Result Analysis (or) Presentation of Result

* It is very important by sharing the knowledge of result or development with others which leads to the various development of test results by other scientist or researchers.

* The steps to be followed for description of test report.
* Statement of the problems
* Materials, methods and procedure used during testing
* Result analysis
* Summary, conclusion and discussion
* Appendices to support findings

### 1. Statement of the Problems

* Statement of the problems describes the objectives of testing which intends about problem.

### 2. Materials, methods and Procedure used during testing

* Materials, methods and Procedure used during testing section includes -

the material to be tested, the conditions of testing specimen, important apparatus used for testing and the major procedure followed by testing which is referenced from the Indian Standard code books.

3. Data presentation and Result analysis

* The result data presented by plotting, it in various methods with proper units assigned.
* In every method of result presentation the statement of result is summarized with the significance of materials.
* The result analysis is done by various methods
* Charts
* Graphs
* Tabulation
* Statement
* Analytic Software

(a) Charts & Graphs

* A chart displays schematic processes based on the outcome and validity.

* Graphs, used to display comparisons between 2 variables.
  Example: line graphs involve an X-axis horizontally and a y-axis vertically on a grid.

(b) Tabulation

* Tabulation is a systematic and logical presentation of numeric data in rows and columns, to facilitate comparison and statistical analysis accompanying with summarizing result.

Major objectives of Tabulation

* To simpilify the complex data
* To bring out essential features of the Data
* To facilitate comparison
* To facilitate statistical analysis
* saving of space

(c) Statement

* Statement Statistics is a form of mathematical analysis that uses quantified models, representations and

synopses for a given set of experimental data or real-life studies.

* Statistics statement studies methodology to gather, review, analyze and draw conclusions from data.

Example: The result of 28 days strength of silicon mixed cube is 25% greater than the conventional concrete.

(d) Analytic software

* Software analysis is the analytics specific to the domain of software systems into account source code, static and dynamic characteristics

Example:-

* BIOVIA MATERIALS STUDIO - Materials studio allows you to easily build, modify, visualize and simulate a wide range of materials.

* LAS X MATERIALS SCIENCE MODULES - LAS X can be enhanced with a range of advanced modules and applications to form a powerful microscopy imaging environment.

* MATLAB - Computation and Plotting

* AUTOCAD - Designing of outline element, 2D and 3D element

* CREO - 3D modeling, Assembly and 2D drafting

* STADD PRO - Designing of structures

* ANSYS - Stress Analysis and Thermal analysis

* ABACUS - Finite element analysis

* ANSYS ELECTRONICS - It is the premier Solution for electromagnetic field, circuit, systems and multi physics simulation ana analysis for electronic design.

4. Summary, conclusion and Discussion

* It describes about the general findings of test or experiment and summarizes the important point.

* Also gives the view about the various error or difficulties occured during testing.
* It gives new views and opinion about material, projected view and acceptability for use in market and environment.

5. Appendices to support findings

* It gives supporting data for testing the materials like code, books, past material testing history and data for better clarity for testing.

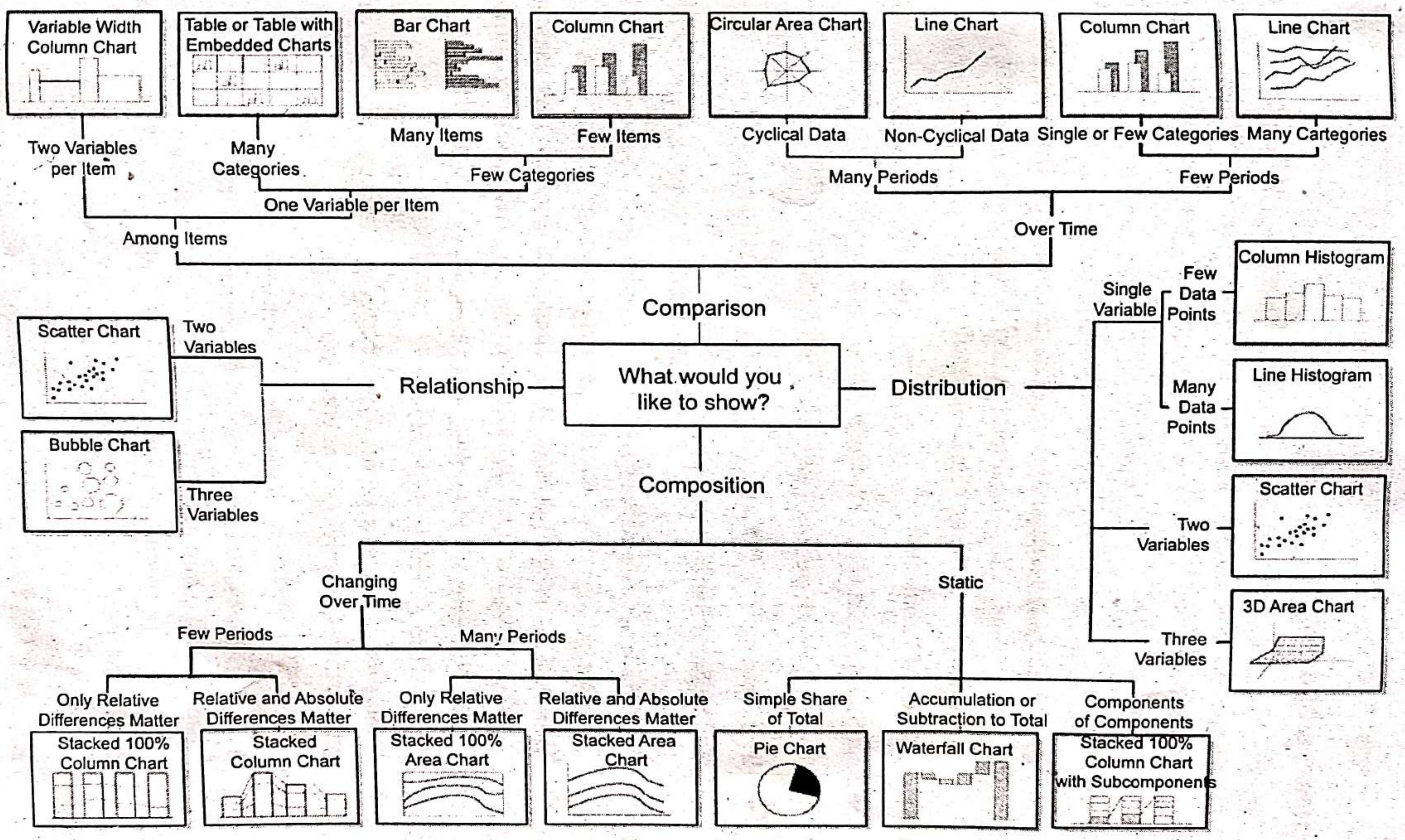



*Fig. 1.13. Types of graphs and charts*

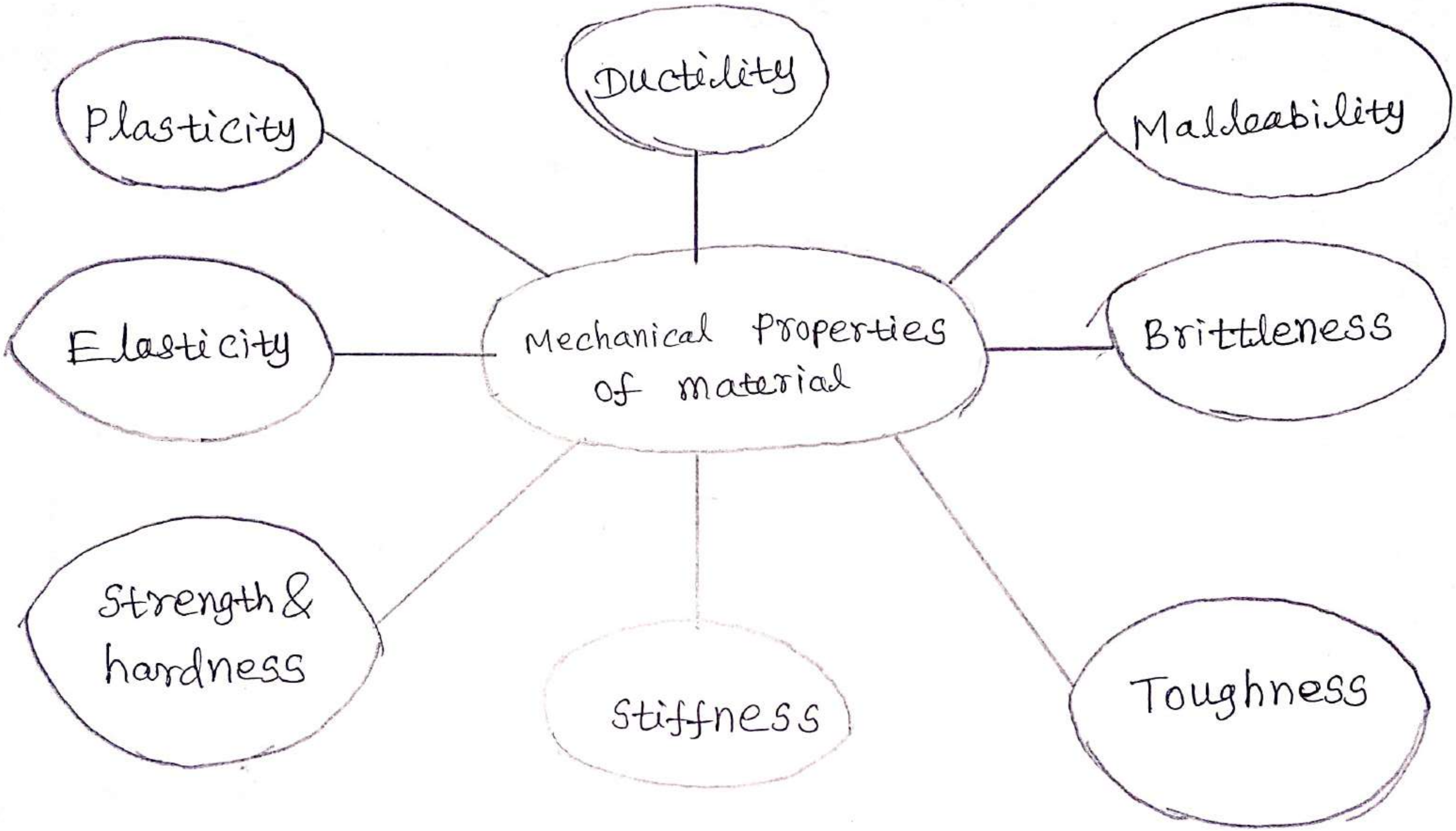
## Introduction to Mechanical Testing

* Mechanical testing reveals the properties of a material under dynamic or static force which is also known as destructive testing.
* Mechanical testing includes methods such as tensile strength, compression strength, impact resistance, fracture toughness and fatigue.
* The study of deformation and fracture in materials is called mechanical behavior of materials.

### 1. mechanical properties of material

#### (i) strength & hardness

* Strength is the ability of a material to resist the externally applied forces without breaking.
* Hardness is a property of resistance to wear, scratching, deformation and machinability, etc.




Mechanical properties

(ii) Stiffness

* Stiffness is the ability of a material to resist deformation under stress.

(iii) Elasticity

* It is the property of a material to regain its original shape after deformation when the external forces are removed.

(iv) Plasticity

* It is the property of a material which retains the deformation produced under load permanently.

(v) Ductility

* Ductility is the property of a material enabling it to be drawn into a wire with the application of a tensile force.
* The ductile material commonly used in engineering practice are mild steel, copper, aluminium, nickel, zinc, tin and lead.

## (vi) Brittleness

* It is the property of breaking of a material with little permanent distortion.
* Cast iron is a brittle material.

## (vii) Malleability

* It is the property of a material to be rolled or hammered into thin sheets.

## (viii) Toughness

* Toughness is the property of a material to resist fracture due to high impact.
* This property is desirable in parts subjected to shock and impact loads.

## (ix) Resilience

* It is the property of a material to absorb energy and to resist shock and impact loads.
* This property is essential for designing the spring materials.

## 2. Property based Testing methods



* The characteristic values obtained from the testing process are used for material development, designing components and in quality assurance.
* There is a range of standard testing methods to characterize the mechanical properties of materials.

| Mechanical Property | Destructive Testing method |
|---|---|
| Elasticity, Plasticity<br>Stiffness, material behavior under static load | Tensile test,<br>compression test,<br>Bending test,<br>Torsion test |
| Creep Behavior | creep rupture test |
| Hardness | Brinell, Rockwell, Vickers |
| Toughness | Impact test<br>Charpy, IZod |
| Fatigue behavior<br>Fatigue Strength | Fatigue test |

## 3. material failure

* Material failure is the loss of load carrying capacity of a material unit.
* The material failure happens due to two major Phenomena,

(a) Deformation failure

(b) Fracture failure

### (a) Deformation failure

* A deformation failure is a change in the physical dimensions or shape of a component that is sufficient for its function to be lost or impaired.

### Plastic deformation & Elastic deformation

* Deformation that appears quickly upon loading can be classed as either elastic deformation or Plastic deformation
* Elastic deformation is recovered immediately upon unloading, whereas Plastic deformation is permanent.

**Creep** It is deformation that accumulates with time. Depending on the magnitude of the applied stress and

* its duration, when deformation may become so large that a component can no longer perform its function.

**Deformation Failure**

- Time Independent
  - * Plastic deformation
  - * Elastic deformation
- Time dependent
  - * Creep deformation

(b) <u>Fracture Failure</u>

* Cracking to the extent that a component is separated into two or more pieces is termed fracture.

**Fracture failure**

- Static Loading
  - * Brittle Failure
  - * Ductile Failure
  - * Environmental Failure
  - * Creep Rupture failure
- Cyclic Loading
  - * High cycle and Low cycle Failure
  - * Fatigue crack growth
  - * Corrosion Fatigue

## Brittle or Ductile fracture:

* The possible growth of the cracks by fatigue, as this can lead to a brittle or ductile fracture once the cracks are sufficiently large.

## Environmental cracking failure

* Fracture may occur as a result of a combination of stress and chemical effects, and this is called environmental cracking failure.

## Corrosion fatigue

* It is the combination of cyclic loading and corrosion. It is often a problem in cyclically loaded.

## Fatigue crack growth

* creep deformation may proceed to the point that separation into two pieces occurs.

## Creep rupture

* The common cause of fracture is fatigue, which is failure due to repeated loading.
* In general, one or more tiny cracks start in the material, and these grow until complete failure occurs.

## High-cycle fatigue & low-cycle fatigue 

* If the number of repetitions of the load is large, say millions then the situation is termed high-cycle fatigue.
* Low cycle fatigue is caused by a relatively small number of cycles; Say tens, hundreds or thousands.
* Low cycle fatigue is generally accompanied by significant amounts of plastic deformation, whereas high-cycle fatigue is associated with relatively small deformations that are primarily elastic.

## Hardness Test

### Hardness

* The hardness is defined as the resistance of a material to permanent or plastic deformation of its surface, usually by indentation, under static or dynamic load.

### Testing methods

* Rockwell hardness test
* Brinell hardness test
* Vickers hardness test

# Brinell hardness test

* The standard method for quantitative evaluation of indentation hardness, which was the widely accepted indentation hardness test, Known as Brinell hardness test.
* The Brinell hardness test consists of forming an indentation by forcing a standard spherical ball indenter into the surface of the material.
* In this test a hardened steel ball of 10 mm in diameter is used as indenter.
* The loading force is in the range of 300 N to 30,000 N.

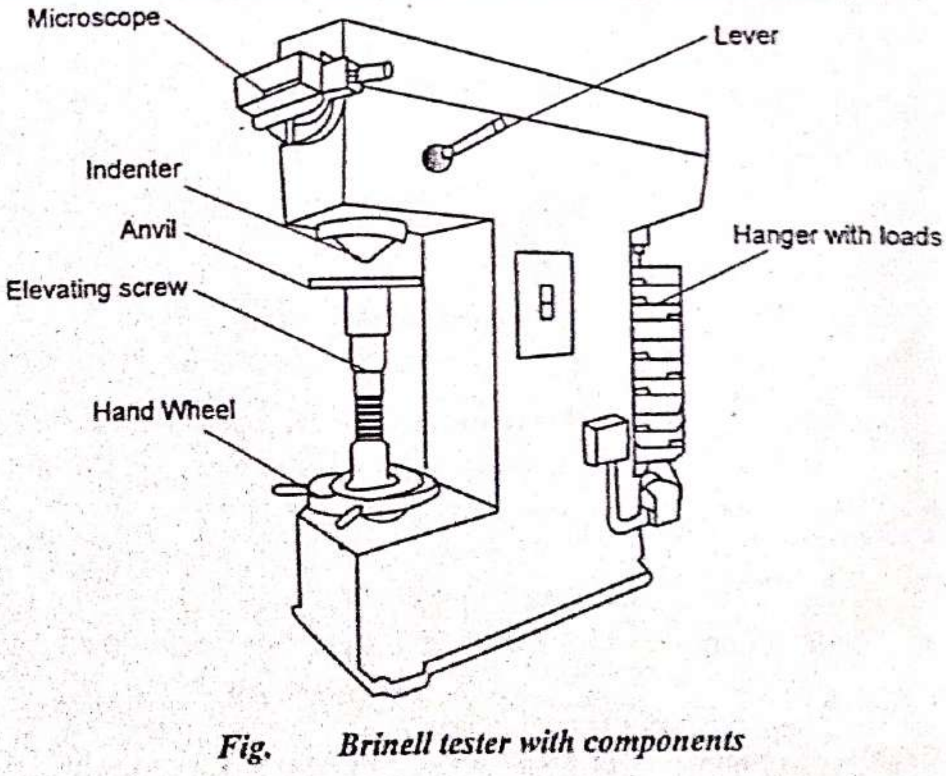



*Fig. Brinell tester with components*

principle

* An indenter (hardened metal ball) is forced into the surface of a test piece and the diameter of indentation 'd' left in the surface after removal of the surface, 'F' is measured under a definite static load applied for a standard period of time.

* The standard Brinell hardness tester operates usually under hydraulic pressure that applies force.

Major components

* Brinell hardness tester
* Brinell microscope
* Indenters and plunger
* Anvil

Indenters

* The diameter of spherical steel ball indenters used in the standard Brinell hardness test is 10 mm.

* The ball indenter normally used is made from heat treated hard high carbon steel.

Load application

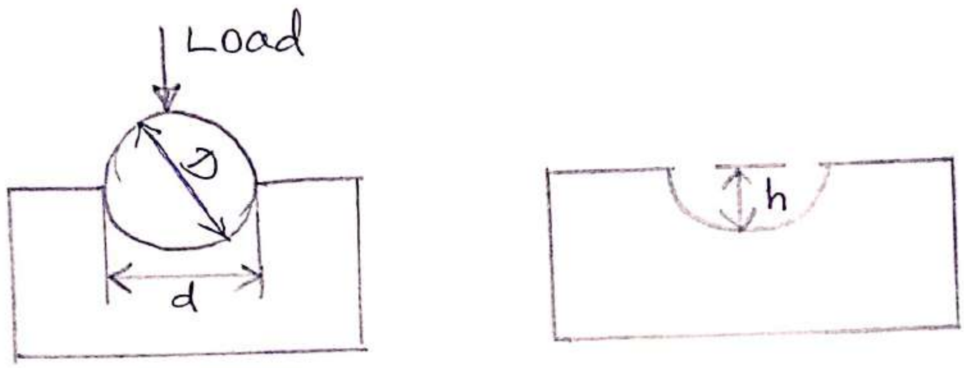
* In Brinell hardness test, load application and time duration is based on the materials..

| Load application | Diameter of ball | Duration | Metals |
|---|---|---|---|
| 3000 Kg | 10 mm | 10 Seconds | Iron, Steel |
| 500 Kg | 10 mm | 30 Seconds | Copper, Annealed Brass, Magnesium alloys, etc. |
| 1000 Kg | 10 mm | 15 Seconds | Gun metal, Bronze and cold worked brass |

## Working

* The surface of the test specimen must be either machined, ground, lapped or polished.
* The specimen is placed on the anvil of the testing machine, and the anvil is raised by rotating the hand wheel so that the specimen surface is brought in tight contact with the apex of the indenter.
* Place the specimen on the test table and apply a minor load to bringing both the pointers on the dial gauge to the 'set' positions.
* Apply the major load on the specimen by turning the loading lever backward.

* Maintain the load on the specimen exactly for the specified dwell time (15 seconds) and then release it by turning the loading lever forwards.
* Take out the specimen and measure the diameter of the indentation formed on it by using the Brinell microscope.




Indenter impression

Brinell hardness number

* The Brinell hardness number (BHN), expressed in units of Kilograms per square millimeter, is defined as the ratio of the applied load (F) to the curved surface area of the elastically recovered indentation.

$$BHN = \frac{F}{Area} = \frac{F}{\frac{\pi D}{2}\left[D - \sqrt{(D^2 - d^2)}\right]}$$

BHN – Brinell Hardness number
D – Diameter of Ball in mm
L – Applied load in Kg
d – Diameter of indentation in mm

| Recommended Materials | Brinell Hardness Number |
|---|---|
| Steel and similarly hard ferrous and other alloys | 140 – 600 |
| Harder Non-ferrous metals and alloys like gun metal / bronze, cold-worked brass | 50 – 200 |
| Non-ferrous metals and alloys like copper, annealed brass, magnesium alloys | 25 – 100 |
| Softer Non-ferrous metals and alloys like aluminium, lead, tin and their alloys | 10 – 50 |

Advantages:-

* It can be used for testing non-homogeneous materials.
* The influence of surface scratches and roughness will be less.
* The Specimen surface can be rough.

* Suitable for hardness tests on forged components, castings and hot-rolled work pieces, etc.

## Disadvantages

* Restriction of application range to a maximum Brinell hardness of 650 HBW.
* Restriction when testing the small and thin-walled specimens.
* The process is slow compared to Rockwell hardness test.
* Due to the larger size of the indentation, the application of Brinell hardness test is not possible on small jobs.

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## Rockwell hardness test

* In the Rockwell hardness test, the depth of the indenter penetration into the specimen surface is measured.
* Each time a test is performed two loads are applied to the sample being tested.

## Principle

* Rockwell hardness test is used to determine the hardness of a metal by 'differential depth' measurement test.

* This hardness testing method involved the measurement of the increment of depth of an indenter forced into the metal by a primary and a secondary load.

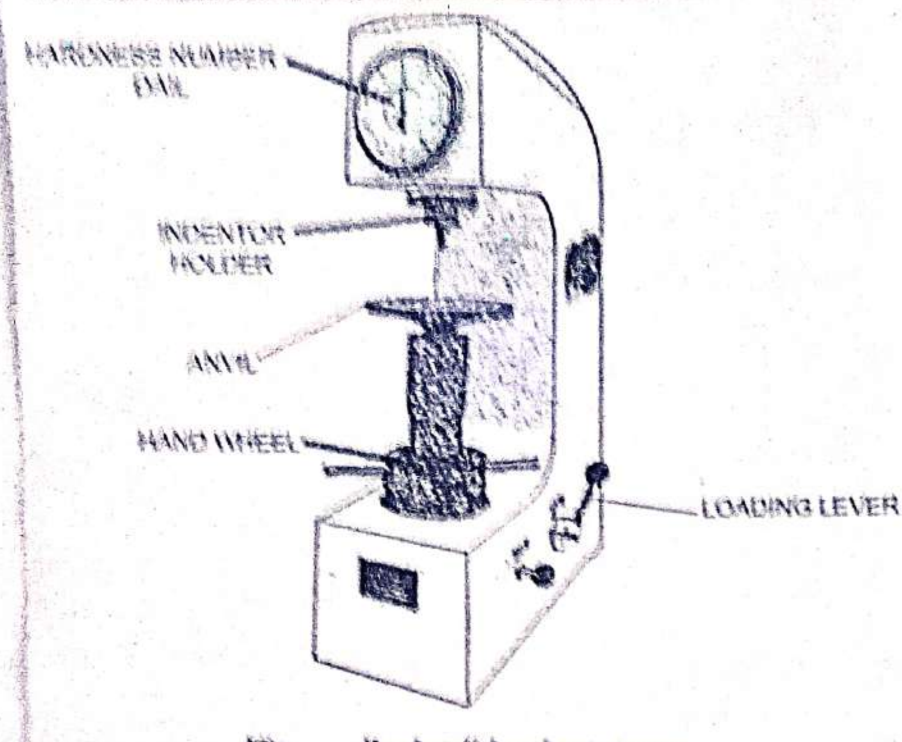



Fig. Rockwell hardness tester

Components

* Rockwell hardness tester
* Indenter

Indenter

* The indenter or penetrator is either made of hardened steel with shape of a spherical ball or made of diamond with shape of a cone having a spherical tip called the 'Brale'.

* The indenter may be either a diameter $1/16''$, $1/8''$ or a spherical diamond cone of $120^\circ$ angle.

Loading condition

* Loads during testing are applied in two stages
  (a) Minor static load
  (b) Major static load

(a) Minor static load

* Minor static load of 10 Kg is applied to form a very shallow indentation on the surface of the specimen through compression of a calibrated coiled spring placed within the machine between the indenter shaft and the dial.
* The purpose of applying minor load is as follows:
  - To eliminate the error that may arise due to variable contacts between the indenter and the surface of the specimen.
  - To set the indenter on the specimen and hold it in position.
  - To reduce the tendency for 'ridging' or 'sinking in' caused by the indenter.

(b) Major Static Load

* Major static load of either 50, 90 or 140 Kg is applied on the surface of the specimen through a system of weights and levers by means of an operating handle in the machine that enlarges the initially formed Indentation under minor load.
* The total static load applied for indentation on the test piece is the summation of the minor load and the major load, which is equal to either 60 or 100 or 150 Kg.

working

* The specimen to be tested is made flat by grinding and then roughly polished because any surface irregularities will be taken care of by the minor load.
* The application of the minor load becomes effective when the surface of the specimen kept on the anvil is brought in contact with the indentor by rotating the anvil elevating wheel.
* First, the indentor is forced into the test material under a preliminary minor load and this depth is recorded.

* with the minor load still applied an additional load is introduced known as the major load which increases the depth of penetration on the sample.
* The major load is then removed, and the force on the sample is returned to the minor load.
* The increase in the depth of penetration that results from applying and removing the major load is used to calculate the Rockwell hardness value.

Rockwell hardness scale

Rockwell hardness scale

| Scale | Indenter | Load(kg) | Dial Number | Typical material |
|---|---|---|---|---|
| A | Diamond cone | Load(kg) 60 | Black | cemented carbide, case hardened surface, thin steel |
| B | 1.5mm ball | 100 | Red | copper, Aluminium, brass, cast iron |
| C | Diamond cone | 150 | Black | Hard cast iron, hardened steel |
| E | 3 mm ball | 100 | Red | Soft Aluminium and alloys, magnesium alloy, bearing metals |
| F | 1.5 mm ball | 60 | Red | Bearing alloy, annealed copper and alloy |

Applications

* It is widely applied in the industry of cemented carbides, copper alloys.
* Thin steel and medium case hardened steel, cast iron, aluminium.

Advantages:-

* High accuracy is achieved.
* Low procurement cost.
* Hardness value is automatically displayed immediately following the indentation process.
* Small size of the impressions produced.
* Generally used for testing of larger samples.
* It can be used for advanced tests.

Disadvantages

* The quality of the indenter and the surface has a strong influence on the test results.
* Sensitivity of the diamond indenter is high. If any damage in diamond indenter, thus producing a risk of incorrect measurements.
* Relatively low sensitivity on the difference in hardness.

## Vickers hardness test

* Vickers hardness test used to determine quantitatively the indentationhardness of material under the application of a constant static load.

## Principle

* A diamond intender in the form of a rigid pyramid with a square base and with a specified angle between opposite faces at the vertex is forced into the surface of a test piece followed by a measurement of the diagonal length of the indentation left in the surface after removal of the test force F.
* The vickers hardness test is a static hardness test method, used for both macro and micro hardness testing. It is an optical method of testing where the size of the indentation left by the indentor is measured to determine the hardness value of a test specimen.

Components:

* Vickers hardness tester
* Indenter

Indenter

* It is made of diamond in the form of a square-based pyramid with an included angle of 136° between opposite faces.

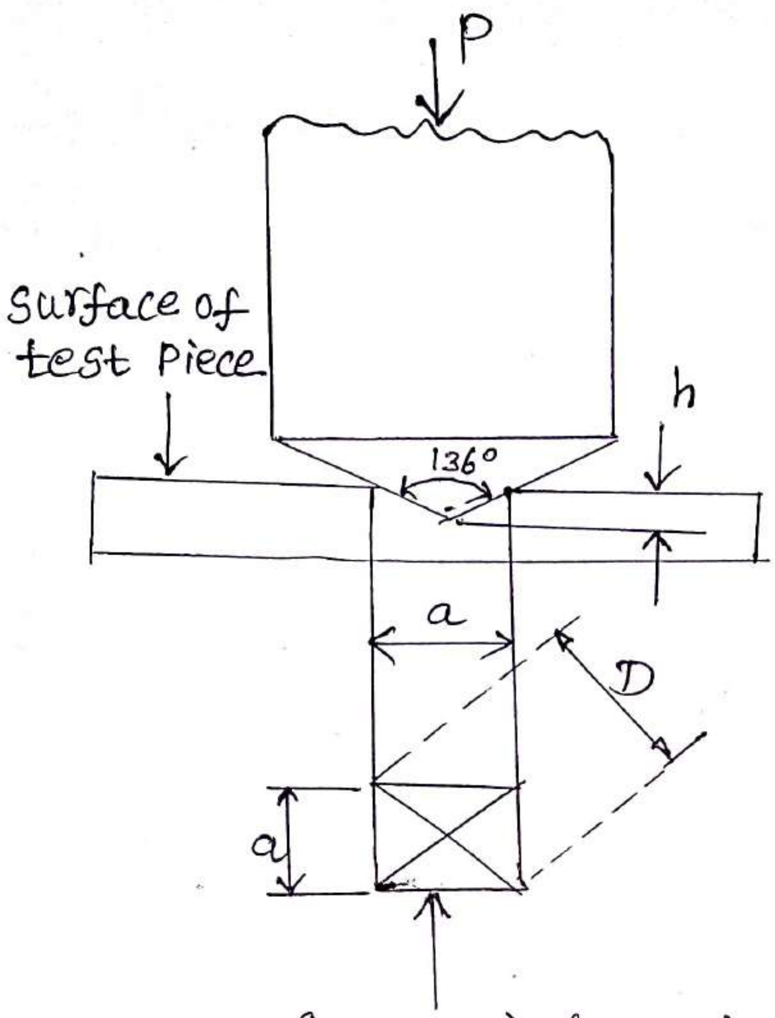
Loading condition

* The loads that can be applied to the indenter in Vickers hardness tester are 1, 2.5, 5, 10, 20, 30, 50, 100 and 120 Kg through appropriate selection of weights.

Working

* Place the specimen carefully on the testing table.
* Turn the hand wheel slowly in the clockwise direction so that the specimen gets focused on the front screen sharply.
* Now bring the inventor to the 'set' position and turn on the loading, dwell unloading cycle.
* The indentation is now projected on the front focusing screen.
* Measure the diagonals along both the axis of the impression and record them.

# Vickers hardness number




Vickers diamond indenter

* The Vickers hardness number (VHN) is defined as the ratio of the applied load, P (Kg) to the surface area of the elastically recovered Pyramid indentation. ($A_s$)

$$VHN = \frac{P}{A_s} \ \frac{Kg}{mm^2}$$

$$VHN = \frac{P}{D^2/1.854}$$

$$VHN = 1.854 \times \frac{P}{D^2}$$

P = Applied load in Kg

$A_S$ = Lateral area of elastically recovered pyramid indentation ($mm^2$)

D = Average diagonal length of square indentation in mm

## Advantages

* Testing thin sheets, small test pieces, thin walled tubes, thin, hard and plated coatings is possible.
* There is only one type of indenter, which can be used for all vickers methods.
* Non-destructive testing is possible.
* The small indentation has no influence on the appearance of tested materials or products.
* Used to find the stress value.

## Disadvantages

* The test location must be prepared.
* Relatively long test time due to the measurement of the diagonal lengths.
* More expensive compare to Rockwell tester.
* Sensitivity of the diamond indentor to damage.

## Tensile Test:-

* Tensile test is a measurement of the ability of a material to withstand forces that tend to pull it apart and to determine to what extent the material stretches before breaking.

## Principle

* A standardized specimen with a known cross section is loaded uniformly with relatively low increasing force in the longitudinal direction.
* A uni axial stress state prevails in the specimen until contraction commences.
* The ratio of stress to strain can be shown from the plotted load-extension diagram.

## Equipment

* Universal Testing machine
* Extensometer
* Scale Vernier Calipers
* Punching tools

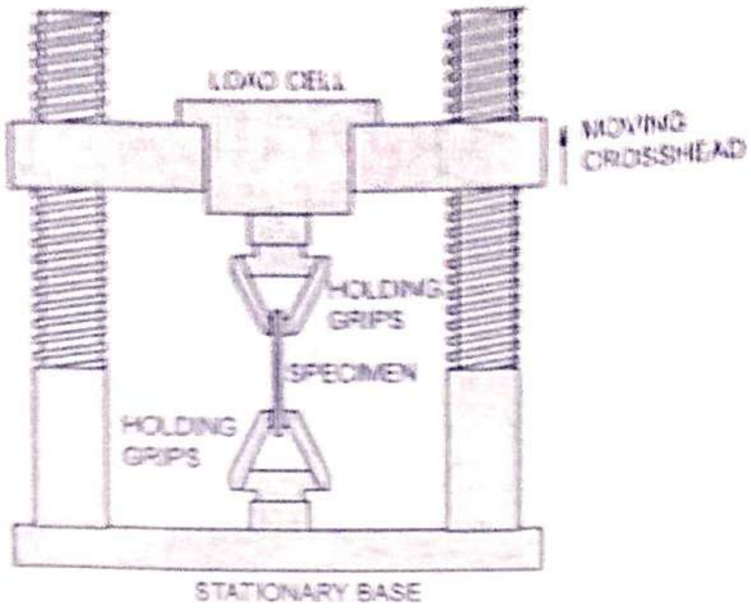



*Fig. Tensile testing apparatus*

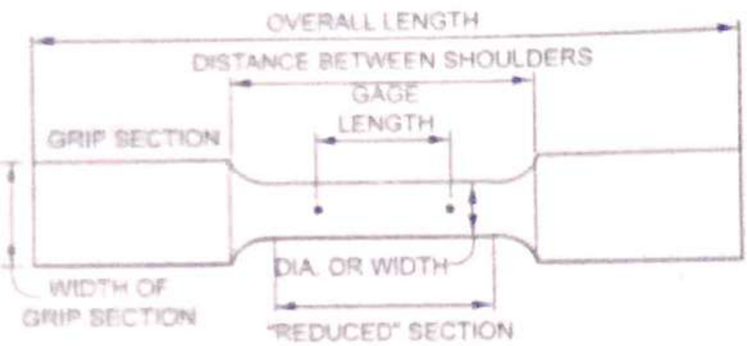



*Fig. Sample Tensile Specimen*

## working

* Initially, the specimen is cleaned and gauge length is marked on it. The ultimate load range to be fixed.
* once the specimen is placed, the jaws are locked.

* Fix the extensometer on the specimen and set the reading to Zero.
* When the specimen is under load, slowly unclamp the locking handle. Note the extension at a convenient load increment.
* Extensometer must be removed before reaching the yield point.
* with the increase in load at some point, the load pointer remains stationary this indicates the yield point.
* with further increase in load, the pointer goes backward and specimen breaks. The load before this breaking is the ultimate load.
* The load at the breaking of the specimen is called as the breaking load.

Advantages

* This test is used to check yield strength, tensile strength and ductile property of material.
* It provides safety and integrity of materials
* It determines batch quality.

Disadvantages

- It is destructive testing, the material gets wasted every time.
- The test is mainly restricted to ductile materials.
- It does not provide information about the material at different temperatures.
- A casting will have different properties than a forging or a sintered metal part.

Impact test

- Impact test is a dynamic test, carried out with notched specimen and known as notched-bar impact test.

Major types

1. Charpy impact test
2. Izod impact test

Purpose of testing

- The purpose of an impact test is to determine the ability of the material to absorb energy during a collision.

* This energy may be used to determine the
  * Toughness
  * Impact strength
  * Fracture resistance

## IZOD and CHARPY TEST

* The charpy v-notch impact test is the most common fracture toughness test.
* A notched specimen is broken by a swinging pendulum and the amount of energy required to break the specimen is recorded.
* The Izod impact test is a standard method of determining the impact resistance of materials.
* A pivoting arm is raised to a specific height and then released.
* The arm swings down hitting a notched sample, breaking the specimen.

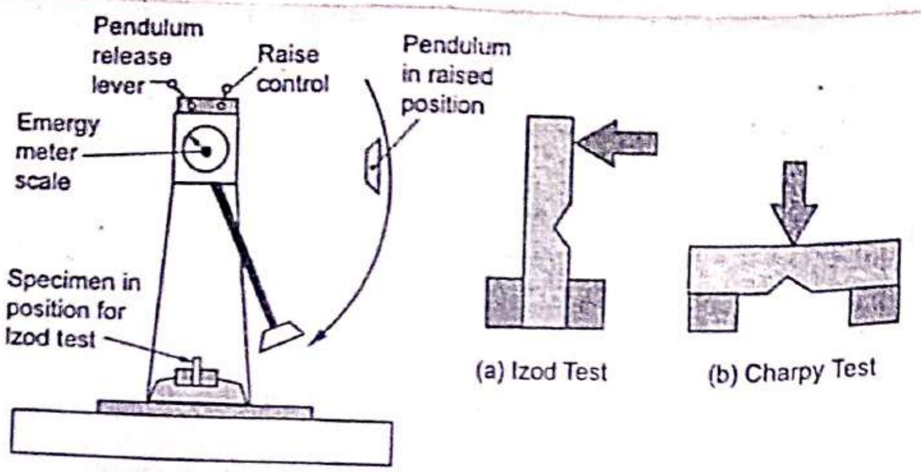



*Fig. Pendulam impact machine and notch position*

## Principle of impact testing

* The sample is placed into a holding fixture with the geometry
* The collision between the weight and specimen generally results in the destruction of the specimen.

## Working

* The test piece is held in the anvil.
* A charpy V-notch specimen is placed across parallel jaws in the impact testing machine
* IZod test specimen is placed perpendicular to jaws.
* The pointer is set upto its maximum value (300J).

* During release, the pendulum strikes and breaks the test piece by a single blow, which consumes part of the energy of the pendulum.
* The energy consumed to break the specimen is measured, usually in Joule.
* Observations of the energy are recorded.

## Applications

* The impact test indicates the notch sensitivity of a material resulting from the presence of internal stress raisers.
* It is used to assess the transition temperature from the ductile to brittle state.

## Advantages

* In expensive
* Higher impact value of a material is higher the toughness value.
* Tests are performed at sub ambient temperatures.
* This impact value can be used to determine the load bearing capacity of a material.

## Disadvantages

* Impact test shows little value for most of the non ferrous metals such as aluminium, copper and their alloys.
* variation in result is improper placement of the test piece in the impact tester.

## Bend Test

* Bending test is standard test method for smooth bars, flat metal spring, concrete, wood, plastics, glass and ceramics.
* It is also called as flexural test.

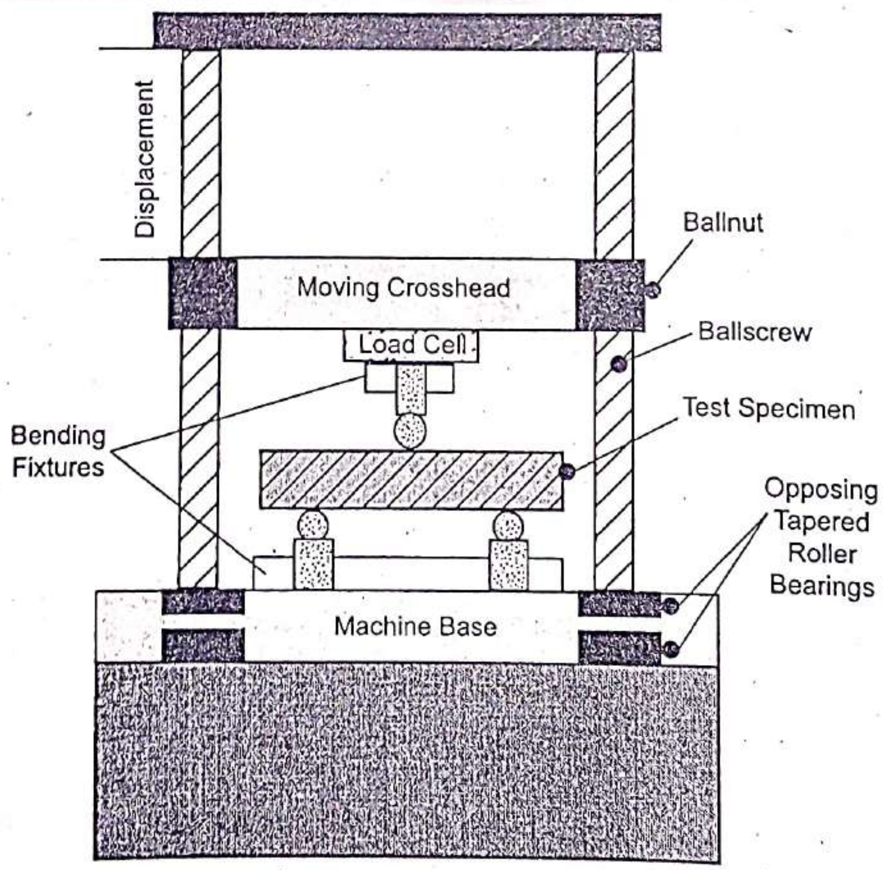



*Fig. Bending test equipment*

## Principle

* Bending tests are conducted by placing a length of material across a span and pushing down along the span to bend the material causing a concave surface.

## Working

* The bending fixture is supported on the platform of hydraulic cylinder of the UTM.
* A loading beam that rests on two rollers on the top of beam to be tested.
* Accurate spacing of the supports and loading points is necessary.
* A load is applied to the loading beam accurately at the mid-point between its two supporting rollers for three point loading.
* The supports are generally knife-edge or convex.
* The load applicator is a rounded knife-edge with an included angle of 60°, applied either at mid span or symmetrically placed from the supports.

* Load and deflection are usually recorded in the test.
* The loading is held in the middle cross head
* At a particular load, the deflection at the center of the beam is determined by using a dial gauge.

## Advantages

* simple geometry.
* Minimum sample machining is required.
* simple test fixture
* It is a simple and inexpensive qualitative test.
* specimen preparation and testing is easy.

## Disadvantages

* The results of the testing method are sensitive to specimen and loading geometry and strain rate.
* More complex stress distributions through the sample.

## Shear test

* In shear test, shear force is the load that causes two parts of the body to slide relative to each other in a direction parallel to their plane of contact.

## Principle

* Shear strength measures a materials ability to resist forces that cause the material to slide against it.
* The specimen is loaded in shear fixtures, load is applied perpendicular to specimen through plunger.
* The phenomenon of shear applies through the shear fixtures is known as shear test.

## Types of shear test

* single shear test
* Double shear test

## Components

* Universal testing machine
* Vernier caliper
* Shear fixtures

working

* The diameter is measured using the vernier caliper.

* Mount the shear fioctures on UTM and load the specimen in shear fixture accordance to need of shear test.

* operate buttons for driving the motor to drive the pump.

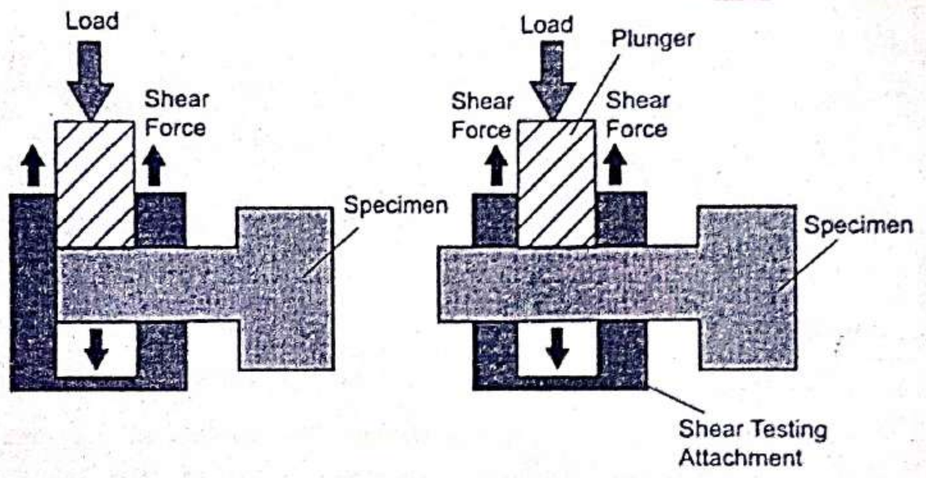



*Fig.* ***Single and double shear loading***

* Gradually move the head control level direction till the specimen shears.

* Take the load at which the specimen shears.

$$\tau = \frac{F}{2A}$$

where $\tau$ - shear stress, F - Force at breaking
A - shearing surface

* In the double shear method, the specimen is sheared off at two cross sections.
* In the single shear process, the specimen only shears away at one cross section.
* The shear strength determined in the shear test is important in the design of bolts, rivets and pins.

## Advantages

* The time of testing is small.
* The adhesion capacity also can be found.
* Result evaluations is direct manner.
* No skilled labour is required.

## Disadvantages

* Due to limitation in diameter of hole, size of testing rod is limited.
* If any error in measurement of the diameter of the specimen gives large variation in results.
* For sheet metal, thickness is restricted.

## Creep test

* In the creep rupture test, a specimen is subjected to load at constant stress and constant temperature.
* This experiment is performed multiple times with different temperature, but always at static loading.
* The plastic deformations are measured in continuous intervals.
* All measured values can be transferred to a creep diagram.
* The measured elongation shows a characteristic curve, which is known as the creep curve.
* The creep rupture test determines the characteristic values for the creep strength and the various strain values.

## Procedure

* Mark the sample for the reduced gauge length.
* Heating chamber is what surrounds the object and maintain the temperature that the object is subjected to gradually

elevated temperature. 

* Measure the dimensions of the given sample
* Fix the ends of the sample up to mark in the jaws of the machine.
* Adjust the extensometer position on the load such that needle on dial is at '0' position.
* The value of change dimension with respect to time for increased temperature with static loading is noted until the specimen fails.
* The final result will be done in following steps.
* calculation of stress
* Plot strain Vs time
* Calculate the creep rate as a function of time and identify the various stages of creep
* Finding the minimum creep rate at each stage

stages of creep

(a) primary creep

* primary creep is the initial creep stage where the slope is rising rapidly at first in a short amount of time.

* After a certain amount of time, the slope will begin to slowly decrease from its initial rise.

(b) secondary creep

* Secondary creep, the creep rate reaches essentially a steady state, in which the creep rate changes little with time.
* This region is approximately constant creep rate.
* During this stage, the steady state is achieved. The creep rate is constant. So, the line on the curve shows a straight line

(c) Tertiary creep

* In this stage, the object creep is continuously increases until the object breaks. During this stage, high stress and high temperature.
* The creep rate is greater and increases continuously till the material undergoes fracture.
* Tertiary creep occurs when the effective cross-sectional area of the specimen is reduced remarkably either due to localized necking.

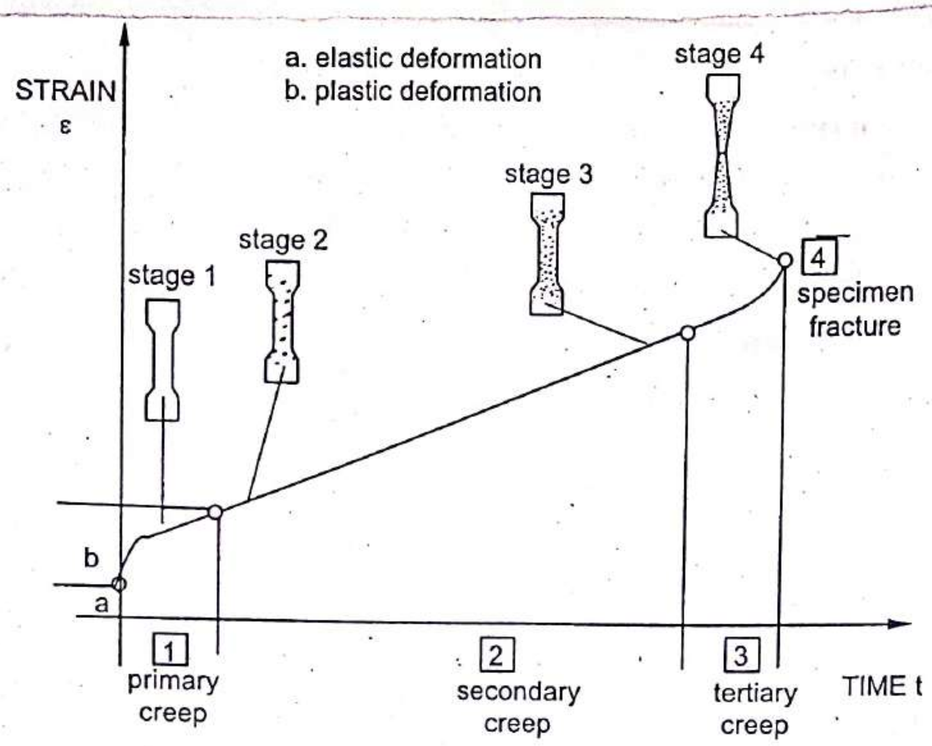



*Fig. Stages of creep*

## Advantages

* To determine the stability of a material and its behaviour when it is put through ordinary stresses like creep test.
* The understanding of their properties and advantages of one material's use over another.

## Disadvantages

* Intermediate stopping of instrument cause error in result.
* The size must be precise.
* It also demands large sample material.

## Fatigue test

* when the component is subjected to repeated cyclic stress leads to failure even through the stress below the yield strength of material.
* This progressive failure of the material at a stress much lower than that required to cause fracture on a single application of load is called a fatigue failure.

## Principle

* A fatigue test is used for the determination of the maximum load that a sample can withstand for a specified number of cycles.
* cyclic fatigue tests produce repeated loading and unloading in tension, compression bending, torsion or combination of these stresses.
* These tests are used to generate fatigue life and crack growth data, identify critical locations. ~~or combinations of these stresses.~~

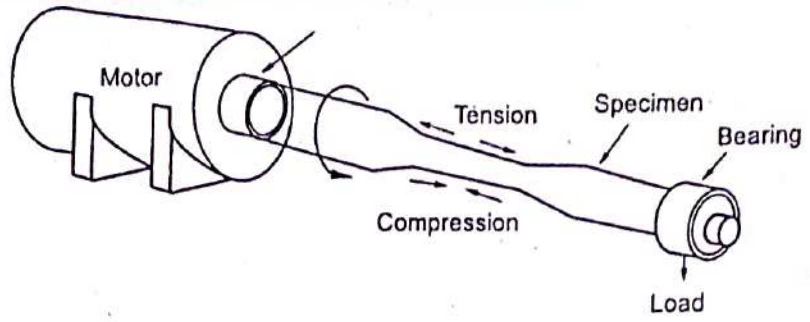



*Fig. .... The cantilever type fatigue testing*

## working

* In fatigue test, a rotating cantilever mounted specimen is subjected to a bending moment.
* In the cylindrical specimen, this creates an alternating stress due to rotary bending.
* After a certain number of load cycles, the specimen fractures because of material fatigue.
* Prepare the specimen as smooth as possible.
* Fit the specimen in the sample holder.
* After fitting the sample, apply the desired load.
* At the top end, the tension is applied and bottom end, compression is provided with rotating of specimen.

* Switch on the instrument to conduct the fatigue test by rotating specimen at 90° with compression and tension and rotate to 180° with same cycle loads.
* Repeat the cycle of stress upto a rotating shaft in four point bending.
* Record the time for the failure, when it occurs.
* Note the appearance of the fractured surface in each case.

## S-N curve

* Fatigue test involve testing the specimens under various cycles of stress, usually in a combination of tension, bending and rotation.
* The test is conducted with variation of stress amplitudes (S); the number of cycles (N), the point of total failure in the specimen is recorded.
* S-N curves are derived from tests on samples of the material to be characterized, where a regular sinusoidal stress is applied by a testing machine which also counts the number of cycles to failure.

* Stress amplitude is defined as the maximum stress, in tension and compression, to which the Specimen is subjected.

* The resulting diagram is called a S-N diagram.

* To determine the S-N curve, a group of fatigue specimens is tested at different stress levels and at each of the several stress levels the loaded specimen is rotated until it fractures.

* The sudden bend of curve indicates the endurance limit.

* The maximum endurance limit for S-N curve is nearly 108 cycles.

* S-N curve calculated in Semi log graph.

High cycle fatigue

* when fatigue tests are conducted with a fixed cycle of load or stress limits, it is called a Stress - Controlled fatigue.

* It is a high cycle fatigue because fatigue failure takes place at high number of Stress cycles, usually more than $10^4$ cycles.

## Low-cycle fatigue

* When fatigue tests are conducted with a fixed cycle of elastic plus plastic strain limits, it is called a strain-controlled fatigue or a low cycle fatigue.
* Fatigue failure takes place when the number of cycles necessary to cause fatigue failure, $N < 10^3$ cycles.
* Testing is conducted with constant strain amplitudes typically at 0.01 – 5 HZ.

## Advantages

* Demonstrate the safety of a structure that may be susceptible to fatigue.
* Generate fatigue data
* Identify critical locations

## Disadvantages

* There is no simple way to relate life predicted by the rule with the characteristics of a probability distribution.
* It does not consider the effect of an overload or high stress which may result in a compressive residual stress that may retard crack growth.

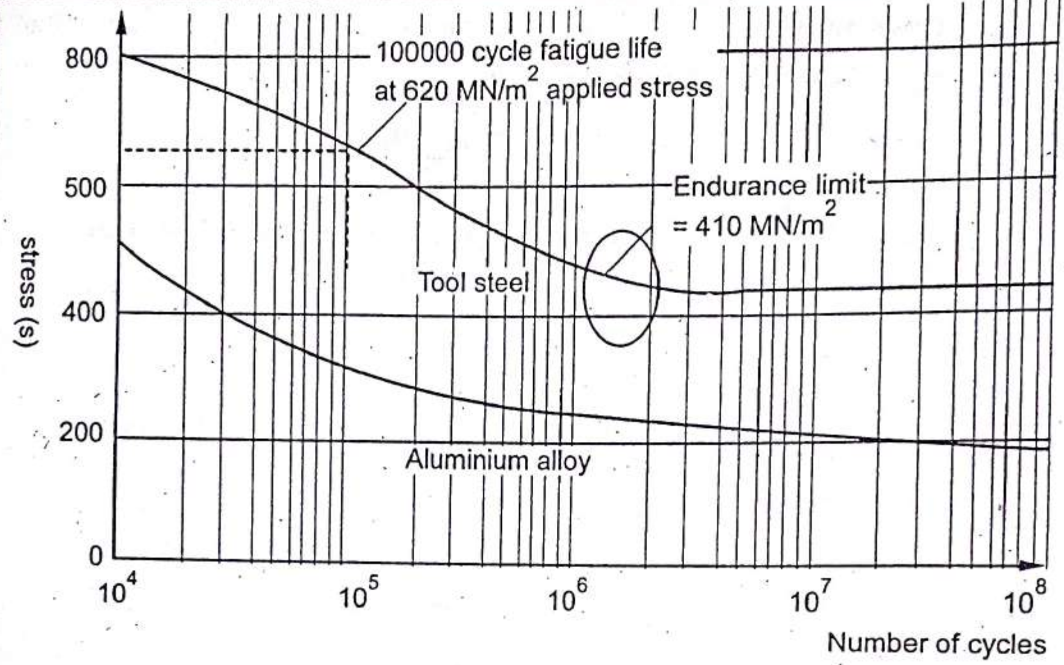



***Fig.*** *S-N curve for steel and aluminum alloy*

UNIT-I Overview of NDT

NDT Versus Mechanical testing:

| Mechanical testing | Non Destructive testing |
|---|---|
| * Test are not made on the components | * Tests are made directly on the components. |
| * 100% testing on object is not Possible | * 100% testing on actual object is possible |
| * An inspection may measure only one or few of the properties | * NDT Can measure many properties |
| * In-service inspection is not Possible | * In service inspection is Possible. |
| * Repeated verifications over a period of time are not possible. | * Repeated verifications overa period of time is possible |
| * cost of test specimen preparation is high | * cost of testspecimen Preparation is very less. |
| * Time conception for inspection is high compare to NDT. | * Rapid inspection is Possible. |
| * All measurements are direct | * Most of the measurements are in-direct |
| * Mechanical Testing gives quantitative measurements. | * NDT gives qualitative measurements |
| * Skilled man power is not required to | * Skilled man power is required to interpret the results |

## Overview of the Non-Destructive Testing methods

### 1. Visual inspection

The most basic NDT method is visual examination. Visual examiners follow procedures that range from simply looking at a part to see if surface imperfections are visible, to using computer controlled camera systems to automatically recognize and measure features of a component. Tools include fiberscopes, borescopes, magnifying glasses and mirrors. Robotic crawlers permit observation in hazardous or tight areas such as air ducts, reactors, pipelines.

**Advantages:**

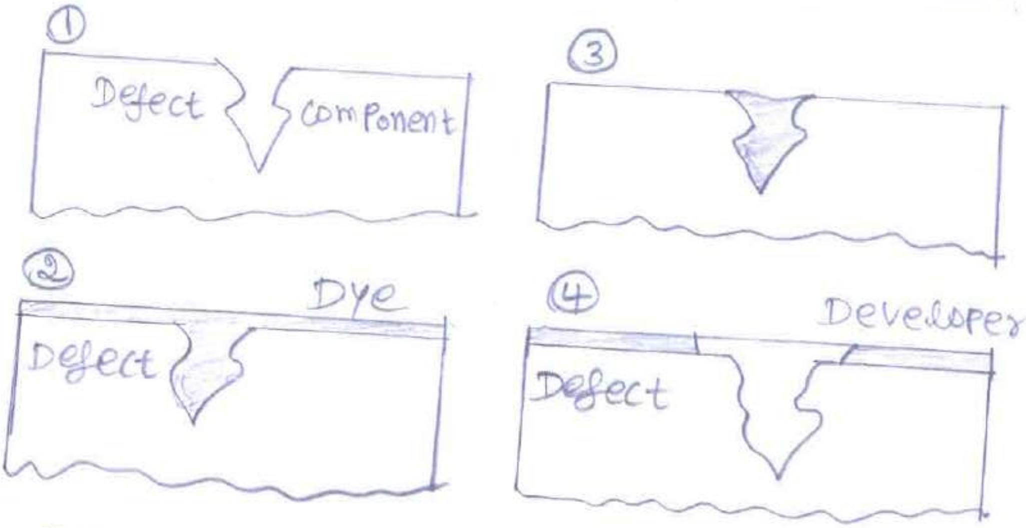
* Simple and usually fast
* Widely used
* Accepted Standard ASTM 02563
* Can pick up a range of defects
* Good for obvious manufacturing flaws

**Limitations:-**

* The inspector should require training and experience
* Must have a good vision
* Internal defects can not be identified
* No permanent records
* Adequate time require for inspection

## Liquid Penetrant testing

The liquid penetrant testing is that when a very low viscosity liquid is applied to the surface of a part, it will penetrate into fissures and voids open to the surface. Once the excess penetrant is removed, the penetrant trapped in those voids will flow back out, creating an indication.




Penetrant testing can be performed on magnetic and non-magnetic materials, but does not work weld on porous materials. Penetrants may be 'visible'.

When performing a inspection, it is imperative that the surface being tested is clean and free of any foreign materials or liquids that might block the penetrant from entering voids or fissures open to the surface of the part. After applying the penetrant, it is permitted to sit on the surface for a specified period of time.

* Then the part is carefully cleaned to remove excess penetrant from the surface.
* When removing the penetrant, the operator must be careful not to remove any penetrant that has flowed into voids.
* A light coating of developer is then be applied to the surface and given time to allow the penetrant from any voids or fissures to the developer, creating a visible indication.
* Then the prescribed developer dwell time, the part is inspected visually, with the aid of a black light for fluorescent penetrants.
* Most developers are fine grained, white talcum like powders that provide a color contrast to the penetrant being used.

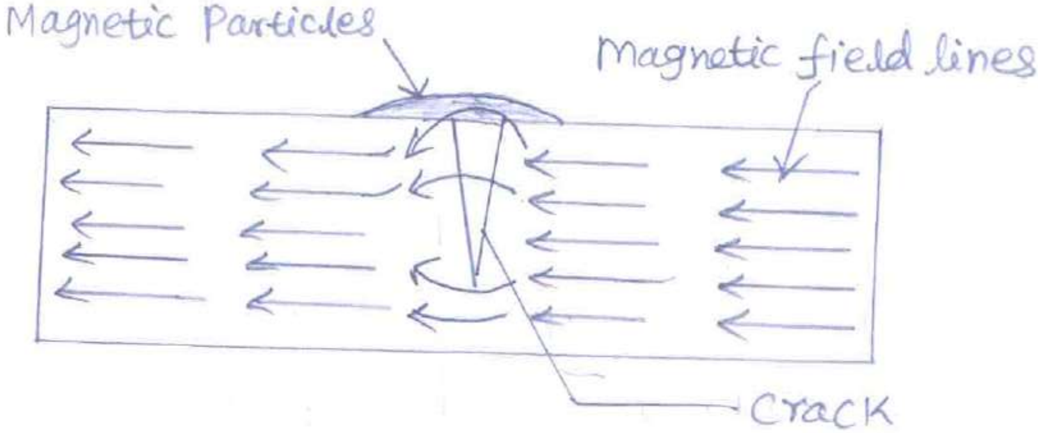
Advantages:

* Relative ease of use.
* Can be used on a wide range of materials.
* Large areas can be inspected rapidly with low cost
* Indications are produced on the surface
* Initial equipment investment is low.

Limitations:

* only detects surface breaking defects
* Requires relatively smooth non porous material.
* Pre cleaning is critical
* Chemical handling precautions necessary.
* Post cleaning is necessary to remove chemicals

## Magnetic Particle Testing




* Magnetic particle testing uses one or more magnetic fields to locate surface and near-surface discontinuities in ferromagnetic materials.
* The magnetic field can be applied with a permanent magnet or an electromagnet.
* When using an electromagnet, the field is present only when the current is being applied.
* When the magnetic field encounters a discontinuity transverse to the direction of the magnetic field, the flux lines produce a magnetic flux leakage field.

* Because magnetic flux lines do not travel well in air, when very fine coloured ferromagnetic particles are applied to the surface of the part the particles will be drawn into the discontinuity, reducing the airgap and producing a visible indication on the surface of the part.
* The magnetic particles may be a dry powder or suspended in a liquid solution, and they may be Colored with a visible dye or a fluorescent dye that fluoresces under an ultravialet light.

Advantages:

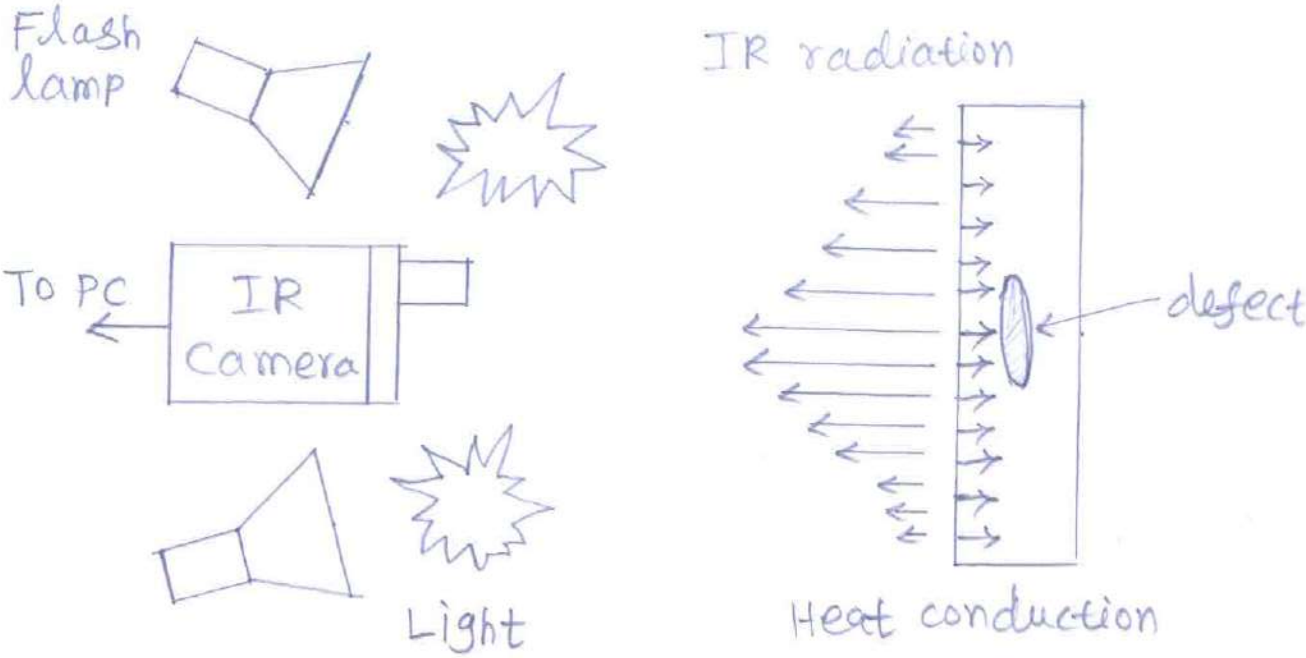
* can detect both surface and near sub-surface defects
* can inspect parts with irregular shapes easily.
* pre-cleaning is not critical
* Indications are visible directly on the specimen surface.
* It is portable and fast method of inspection

Limitations:-

* cannot inspect non-ferrous materials
* special Power require for inspection of large parts.
* Some parts may require removal of

* Limited subsurface discontinuity detection capabilities.
* Alignment between magnetic flux and defect is important.

## Thermography Testing




* Thermography testing is used to measure the surface temperatures based on the infrared radiation given off by an object as heat flows through, to or from that object.
* The majority of infrared radiation is longer in wave length than visible light but can be detected using thermal imaging devices, commonly called infrared cameras.
* For accurate IR testing, the part being investigated should be in direct line of sight with the camera.

* Thermal imaging can be used to detect corrosion damage, de-laminations, debonds, voids, inclusions as well as many other detrimental conditions.

Advantages:

* Non contact
* Can work at a distance
* Fast and reliable
* Portable

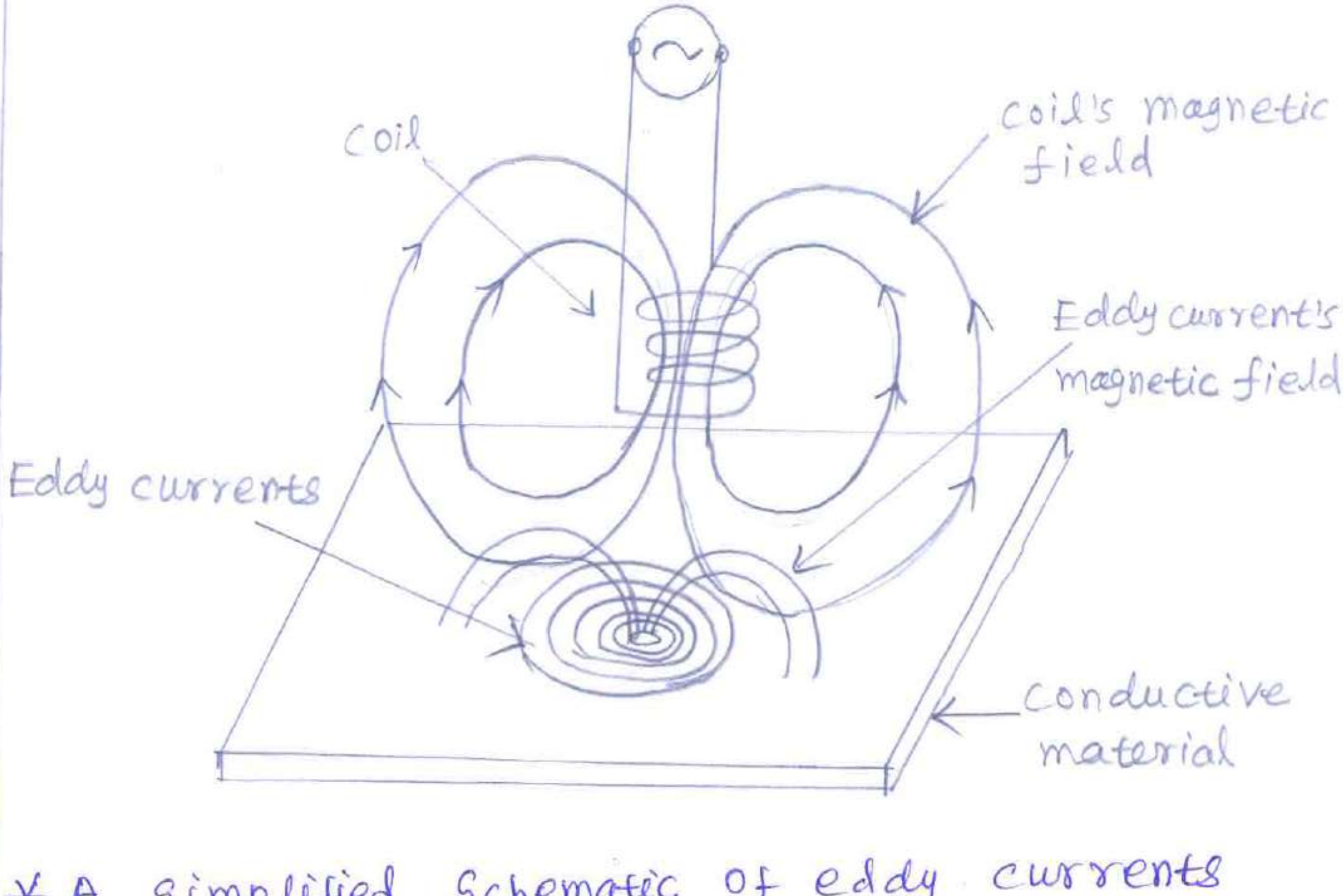
Limitations

* Non intrisically safe.
* There must be a temperature difference for certain surveys.
* Operator experience is essential
* Filters may be needed for certain applications
* Sensitivity and resolution reduce with distance and angle of view

Eddy Current Testing

* Eddy current testing uses the fact that when an alternating current coil induces an electromagnetic field into a conductive test piece, a small current is created around the magnetic flux field, much like a magnetic field is generated around an electric current.

* The flow pattern of this secondary current, called an eddy current, will be affected when it encounters a discontinuity in the test piece, and the change in the eddy current density can be detected and used to characterize the discontinuity causing that change.




* A simplified schematic of eddy currents are generated by an alternating current coil.
* By varying the type of coil, this test method can be applied to flat surfaces or tubular products.
* This technique works best on smooth surfaces and has limited penetration, usually less than 1/4".

* Encircling coils are used to test tubular and bar - shaped products.
* The tube or bar can be fed through the coil at a relatively high speed, allowing the full cross-section of the test object to be interrogated.
* However, due to the direction of the flux lines, circumferentially oriented discontinuities may not be detected with this application.

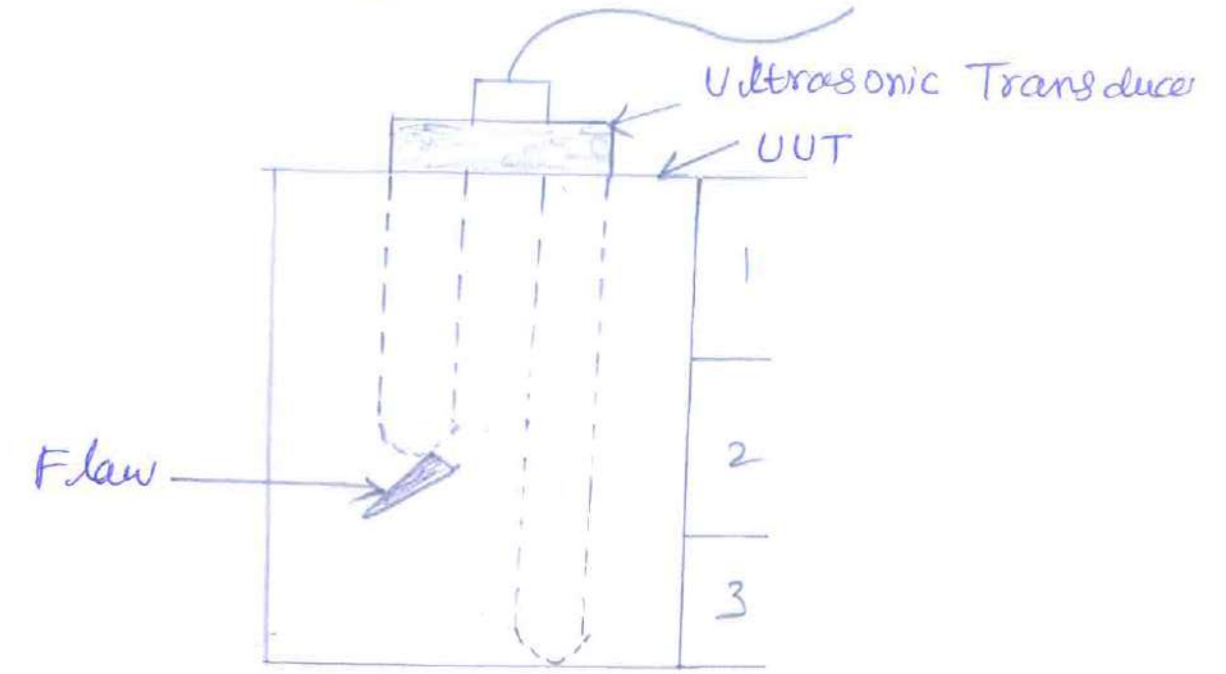
Advantages:

* Sensitive to small cracks and other defects.
* Detects surface and near surface defects
* Inspection gives immediate results.
* Equipment is very portable
* minimum part preparation is required.

Limitations:

* only conductive material can be inspected.
* Surface must be accessible to the probe.
* Skill and training required.
* Depth of penetration is limited.
* Reference standards needed for setup.

# Ultrasonic Testing




* Ultra high frequency sound is introduced into the part being inspected and if the sound hits a material with different acoustic impedance, some of the sound will reflect back to the sending unit and presented on a visual display

* By knowing the speed of the sound through the part and the time required for the sound to return the sending unit, the distance to the reflector can be determined.

* The most common sound frequencies used in UT between 1.0 and 10.0 MHZ, which are too high to be heard and do not travel through air.

* The lower frequencies have greater penetrating power but less sensitivity, while the higher frequencies don't penetrate as deeply but can detect smaller

* sound is introduced into the part using an ultrasonic transducer that converts electrical impulses from the UT machine into sound waves, then converts returning sound back into electric impulses that can be displayed as a visual representation on a digital or LCD screen.

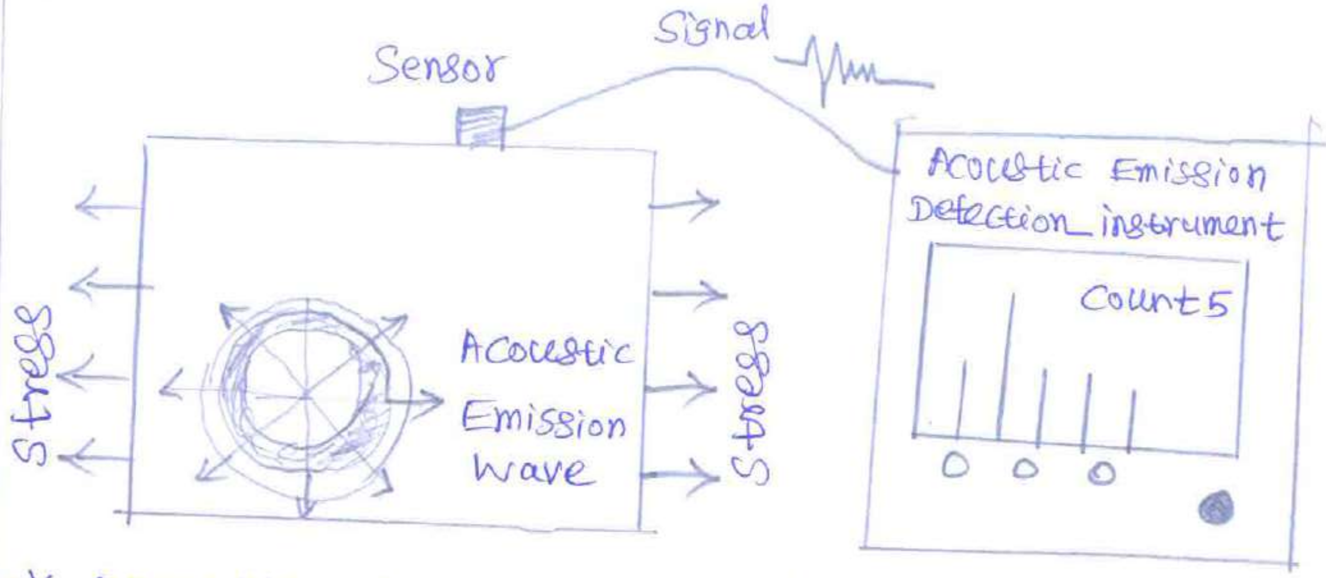
Advantages:-

* position, size and type of defects can be determined.
* Instant test results.
* portable
* Capable of being fully automated
* Access to only one side necessary.

Limitations:-

* Surface must be accessible to transmit ultrasou
* Skill and training is more extensive
* A coupling medium required to promote transfer of sound.
* Materials that are rough, very small are difficult to inspect.
* Cast iron and other Coarse grained materials are difficult to inspect.

## Acoustic Emission Testing:-




* Acoustic Emission Testing is performed by applying a localized external force such as an abrupt mechanical load or rapid temperature or pr. change to the part being tested.

* The resulting stress waves in turn generate short lived, high frequency elastic waves in the form of small material displacements or plastic deformation, on the part surface that are detected by sensors that have been attached to the part surface.

* when multiple sensors are used, the resulting data can be evaluated to locate discontinuities in the part.

Advantages :-

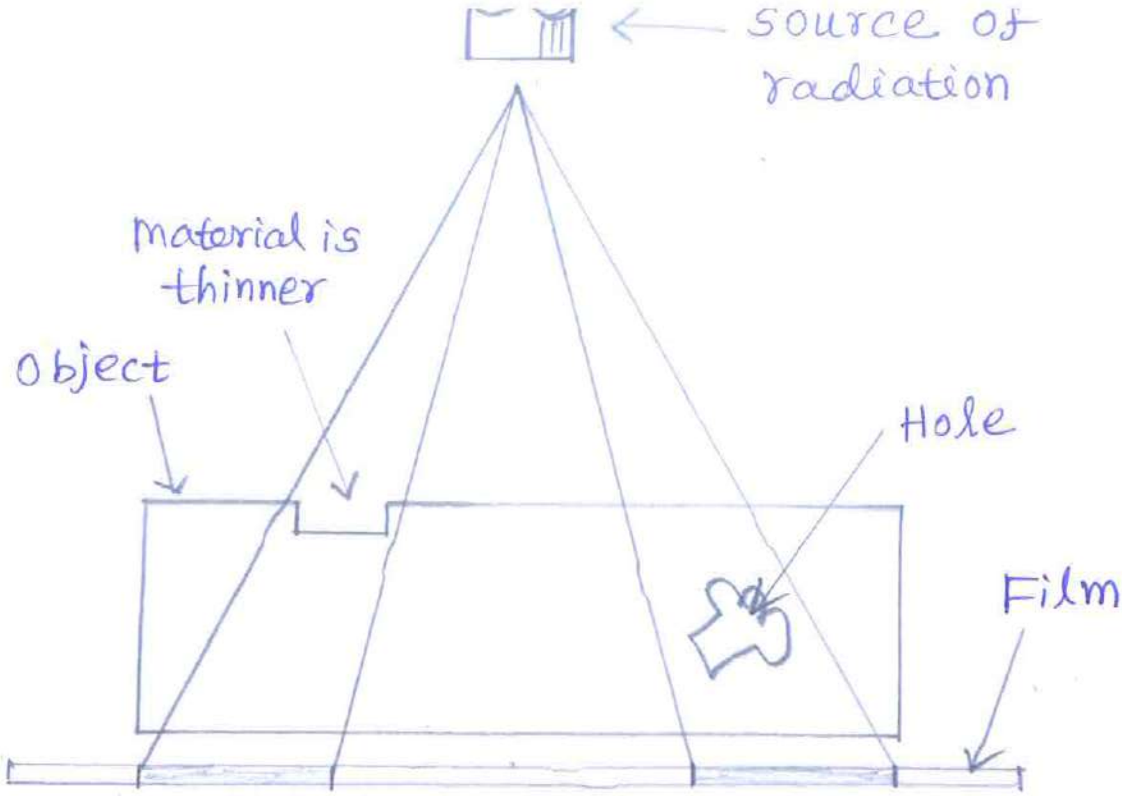
* High sensitivity
* Early and rapid detection of defects, flaws, crack etc.
* Real time monitoring
* cost reduction
* No need for scanning the whole structure surface.

Limitations :-

* It is stress unique and each loading is different.
* Loading history to be maintained.
* Acoustic Emission can be subjected to noise
* The structure under test will weakened the acoustic stress wave.

Radiography Testing

* Industrial radiography involves exposing a test object to penetrating radiation so that the radiation passes through the object being inspected and a recording medium placed against the opposite side of that object.




* For thinner or less dense materials such as aluminium, electrically generated X-radiation (X-rays) is commonly used. Thicker or denser materials and gamma radiation is generally used.

* Gamma radiation is given off by decaying radioactive materials, with the two most commonly used sources of gamma radiation being Iridium-192 and Cobalt-60.

* IR-192 is generally used for steel upto 2.5 to 3 inches, depending on the curie strength of the source.

* Co-60 is usually used for thicker materials due to its greater penetrating ability.

* The recording media can be industrial X-ray film or one of several types of digital radiation detectors.
* Darker areas, more radiation has passed through the part and lighter areas, less radiation has penetrated.
* If there is a void or defect in the part, more radiation passes through, causing a darker image on the film or detector

Advantages:-

* Technique is not limited by material type or density
* Can inspect assembled components.
* minimum surface preparation required
* Surface and sub surface defects can be detected.
* Provides a permanent record of the inspection

Limitations:-

* Many safety precautions for the use of high intensity radiations.
* Many hours of technician training prior to use.
* Access to both sides of sample required.
* orientation of equipment and flaw can be critical.
* Expensive initial equipment cost.

various Physical characteristics of materials and their applications in NDT

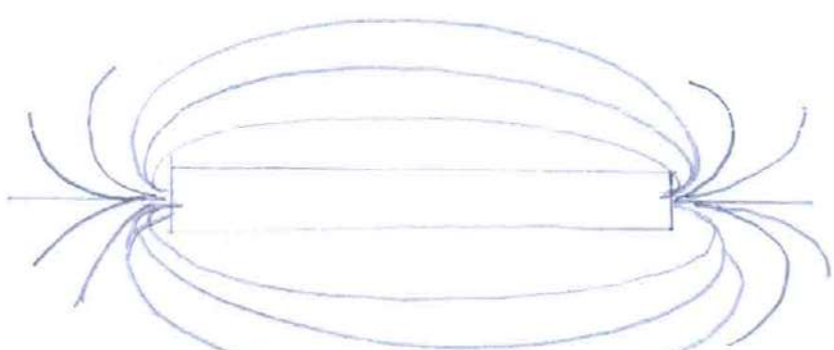
Heat Transfer:-

* Heat transfer is the exchange of thermal energy between physical systems..
* The rate of heat transfer is dependent on the temperatures of the systems and the properties of the intervening medium through which the heat is transferred.
* The three fundamental modes of heat transfer are conduction, convection and radiation.
* Heat transfer, the flow of energy in the form of heat, is a process by which a system's internal energy is changed.
* The direction of heat transfer is from a region of high temp. to another region of lower temperature, and is governed by the second law of thermodynamics.
* Heat transfer will occur in a direction that increases the entropy of the collection of systems.
* Thermal equilibrium is reached when all involved bodies and the surroundings reach the same temperature.
* Thermal expansion is the tendency of matter to change in volume in response to a change in temperature.

* The basic premise of thermographic NDT is that the flow of heat from the surface of a solid is affected by internal flaws such as dis-bonds, voids or inclusions.
* The use of thermal imaging systems for industrial NDT applications will be the focus of this material.

Magnetism :-

* Magnetism is a class of physical phenomena that are mediated by magnetic fields.
* Electric currents and the magnetic moments of elementary particle give rise to a magnetic field, which acts on other currents and magnetic moments.
* Every material is influenced to some extent by a magnetic field.
* The most familiar effect is on permanent magnets, which have persistent magnetic moments caused by ferromagnetism



* The prefix ferro- refers to iron, because permanent magnetism was first observed in a form of natural iron ore called magnetite, $Fe_3O_4$.
* Most materials do not have permanent moments. Some are attracted to a magnetic field. others are repulsed by a magnetic field. Others have a more complex relationship with an applied magnetic field.
* The magnetic state of a material depends on temperature and other variables such as pressure and the applied magnetic field. A material may exhibit more than one form of magnetism as these variables change.
* Magnetism is used to inspect material for flaws.
* One of the NDT methods commonly used is called magnetic particle inspection.
* The reason for use this test is to find small defects in objects before they become bigger defects and cause serious problems

* In magnetic particle inspection, a magnet or electrical current is used to establish a magnetic field in the object.
* The fillings should align along the magnetic lines of force.
* If a crack or other defect is present, the magnetic fines of force will be disrupted and the magnetic particles will cluster along the edges of the flaw.

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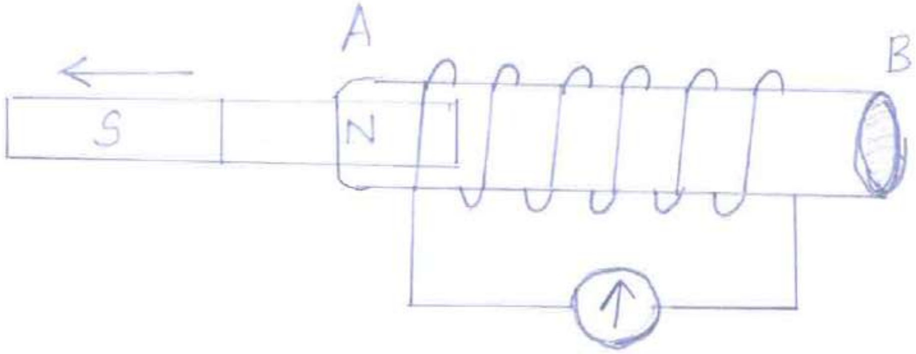
Eddy current principle:

The eddy current method is based on the principle of generating circular electrical currents in a conductive material This is achieved by the use of a coil connected to an alternating current generator driving an alternating magnetic field.

ECT is one of many electromagnetic testing methods used in nondestructive testing (NDT) making use of electromagnetic induction to detect and characterize surface and sub-surface flaws in conductive materials.

Electrical induction:




* Eddy currents are loops of electrical current induced within conductors by a changing magnetic field in the conductor, due to Faraday's law of induction.

* Eddy currents flow in closed loops within conductors, in planes perpendicular to the magnetic field.

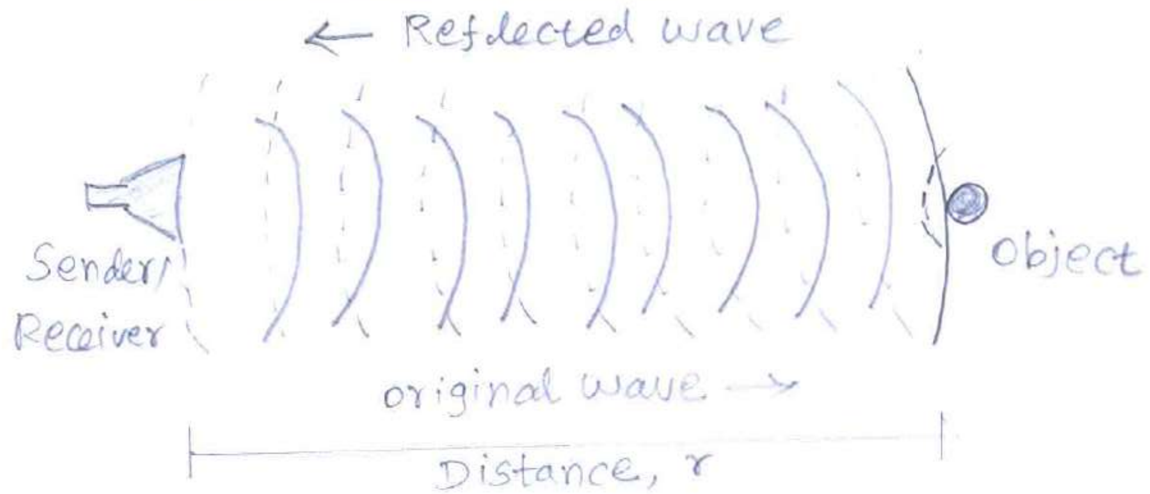
* The magnitude of the current in a given loop is proportional to the strength of the magnetic field, the area of the loop, the rate of change of flux, and inversely proportional to the resistivity of the material.

* Eddy currents are also used in heat objects in induction heating furnaces and equipment, and to detect cracks and flaws in metal parts using eddy-current testing instruments.

* Eddy current testing is used to detect surface and near surface flaws in conductive materials.

* Eddy current inspection is also used to sort materials based on electrical conductivity and magnetic permeability and measures the thickness of thin sheets of metal and non conductive coatings such as paint.

<u>Ultrasound Frequency</u>




* Ultrasounds are sound waves with frequencies higher than the upper audible limit of human hearing. This limit varies from person to person and is approximately 20,000 Hertz in healthy young adults.

* Ultrasound devices operate with frequencies from 20 KHZ upto several gigahertz.

* Ultrasonic testing is a type of non-destructive testing commonly used to find flaws in materials and to measure the thickness of objects.
* Frequencies of 2 to 10 MHZ are common but for special purposes other frequencies are used.
* Inspection may be manual or automated and is an essential part of modern manufacturing processes.
* Most metals can be inspected as well as plastics and aerospace composites.
* Lower frequency ultrasound (50-500 KHz) can also be used to inspect less dense materials such as wood, concrete and cement.

## Acoustics

* Acoustics is the interdisciplinary science that deals with the study of all mechanical waves in gases, liquids and solids including topics such as vibration, sound, ultrasound and infrasound.
* The study of acoustics revolves around the generation, propagation and reception of mechanical waves and vibrations.

* Acoustics look first at the pressure levels and frequencies in the sound wave and how the wave interacts with the environment
* This interaction can be described as either diffraction, interference or a reflection or a mix of the three.

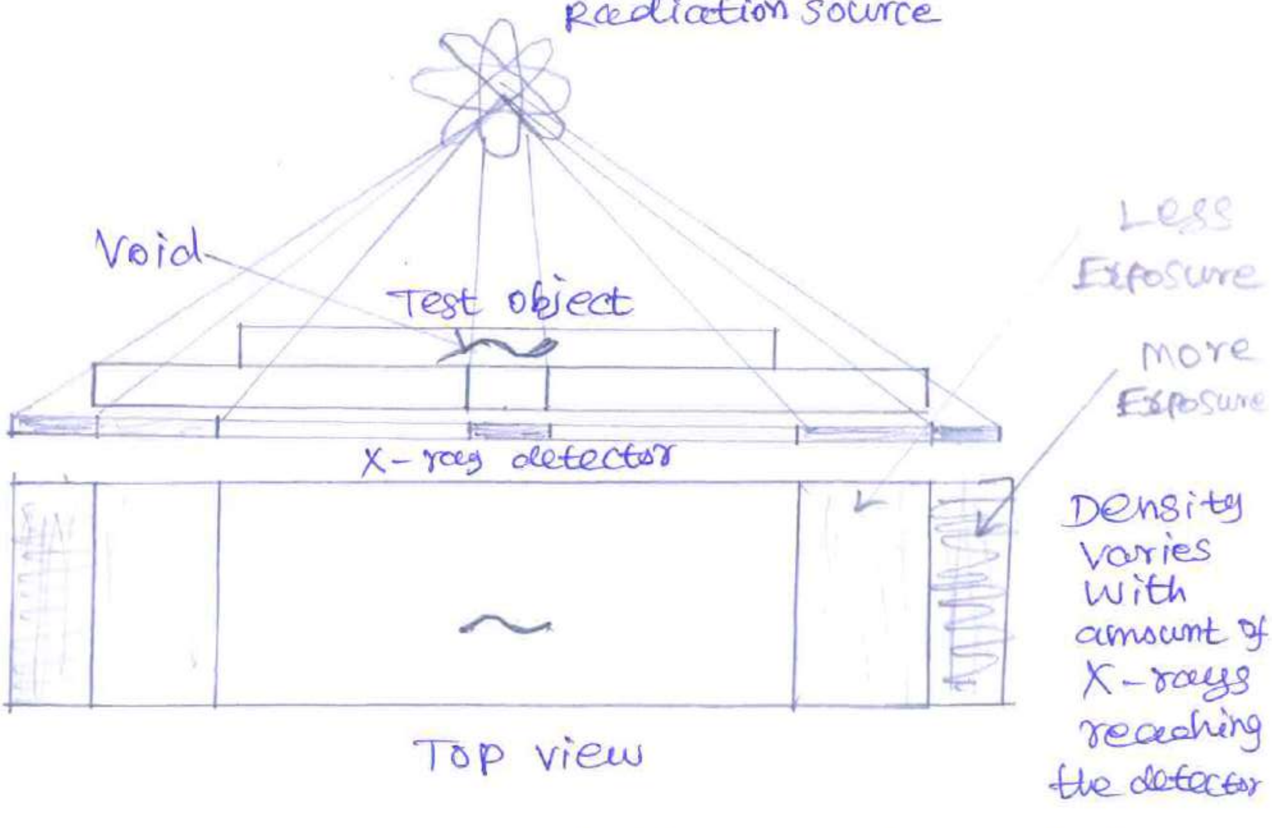
| | | | | |
|---|---|---|---|---|
| Cause | Genarating mechanism (transduction) | Acoustic wave Propagation | Reception (Transduction) | Effect |

* Acoustic emission is a very versatile, non-invasive way to gather information about a material or structure. It is applied to inspect and monitor pipelines, pressure vessels, storage tanks, bridges, aircraft, bucket trucks, variety of composite and ceramic composites.
* It is also used in process control applications such as monitoring welding processes.

## Radiography

* Radiography is an imaging technique that uses electromagnetic radiation other than visible light, especially X-rays, to view the internal structure of a non-uniformly composed and opaque object.

* To create the image, a heterogeneous beam of X-rays is produced by an x-ray generator and is projected toward the object.
* A certain amount of X-ray is absorbed by the object, which is dependent on the particular density and composition of that object.
* X-rays pass through the object was captured behind the object. by a detector.
* The detector provide a superimposed 2D representation of all the object's internal structures.




* X-rays and gamma rays differ only in their source of origin.
* X-rays are produced by an X-ray generator and gamma radiation is the product of radioactive atoms.

* They are both part of the electro magnetic Spectrum.
* They are wave forms, as are light rays, microwaves and radio waves.
* X-rays are used to produce images of objects using film or other detector that is sensitive to radiation.
* The test object is placed between the radiation source and detector.
* This variation in radiation produces an image on the detector that often shows internal features of the test object.
* Radiography is used to inspect almost any material for surface and subsurface defects.
* X-rays also used to locates and measures internal features, confirm the location of hidden parts in an assembly, and to measure thickness materials.

Visual inspection:

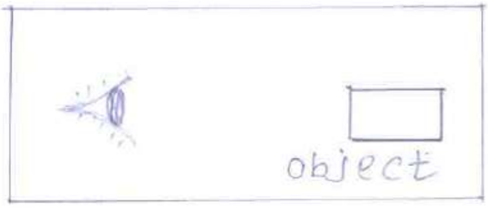
* Visual testing inspections may be by direct viewing, using line of sight vision or may be enhanced with the use of optical instruments such as magnifying glasses, mirrors, boroscopes and computer assisted viewing systems.

* Corrosion, misalignment of parts, physical damage and cracks are just some of the discontinuities that may be detected by visual examinations.

* visual inspection is classified into two categories:

1. Unaided inspection
2. Aided inspection

Unaided visual inspection:




* There is no interruption between the eye and the object is called Unaided visual inspection.
* The human eye is one of mankind's most fascinating tools.
* It has greater precision and accuracy than many of the most sophisticated cameras.
* It has unique focusing capabilities and has the ability to work in conjunction with the human brain so that it can be trained to find specific details or characteristics in a part or test piece.

* The human eye is capable of assessing many visual characteristics and identifying various types of discontinuities.
* The eye can perform accurate inspections to detect size, shape, color, depth, brightness, contrast and texture.
* In the fabrication industry, weld size, contour, length and inspection for surface discontinuities are routinely specified.
* Forgings and castings are normally inspected for surface indications such as laps, seams and other various surface conditions.

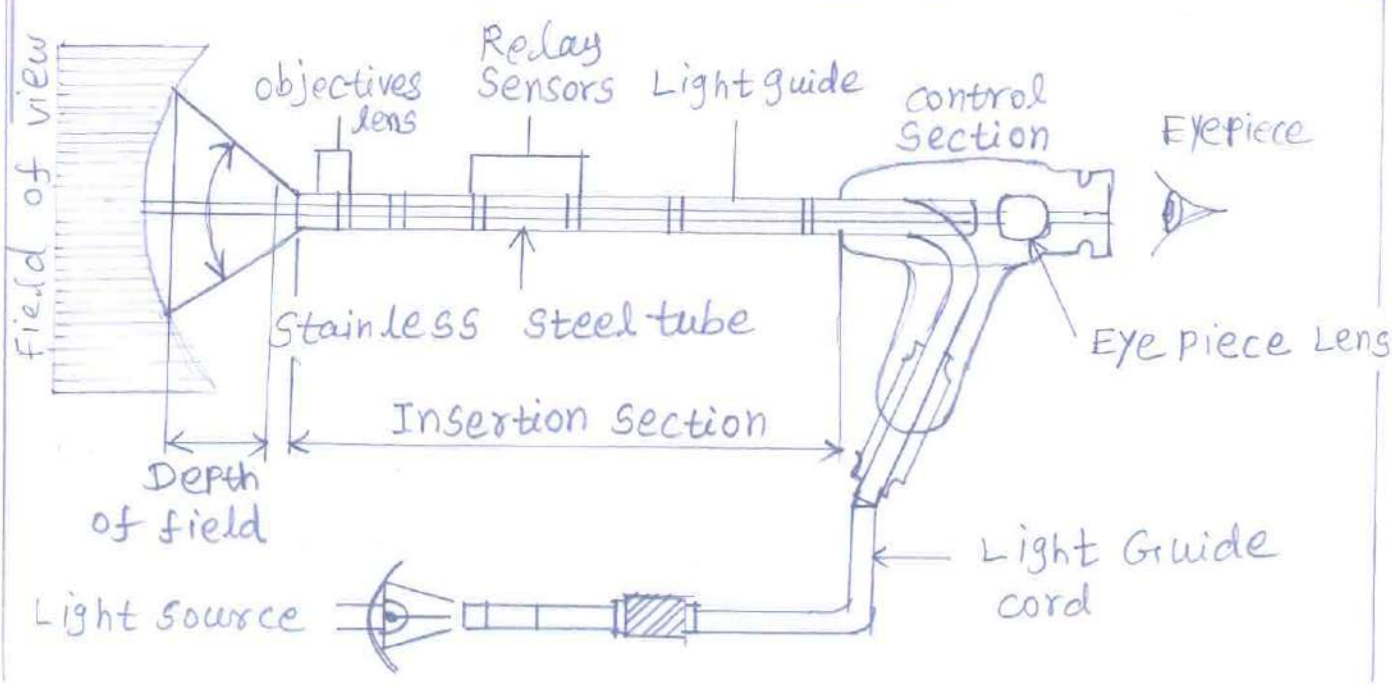
## Aided visual inspection:

* It is the process of examination and evaluation of systems and components by use of optical aids. The following list includes most optical aids currently in use.
* mirrors
* Magnifying glasses, multilens magnifiers, measuring magnifiers
* Microscopes (optical, electron)
* optical flats (for surface flatness measurement)

* Borescopes and fiber-optic borescopes
* optical comparators
* Photographic records
* closed-circuit television systems
* Machine vision systems

## Borescope:

* A Borescope is an optical device consisting of a rigid or flexible tube with an eyepiece on one end, an objective lens on the other end linked together by a relay optical system.
* The optical system is surrounded by optical fibers used for illumination of the remote object.
* An internal image of the illuminated object is formed by the objective lens and magnified by the eyepiece which present's it to the viewer's eye.



* Borescopes are commonly used in the visual inspection of aircraft engines, indegtrial gas turbines, steam turbines, diesel engines, automotive and truck engines.
* Gas and steam turbines require particular attention because of safety and maintenance requirements.
* Borescope can be used to inspection of engines to prevent unnecessary maintenance.
* They are also used to inspect machined and cast parts critical interior surfaces such as burrs, surface finish or complete through-holes.

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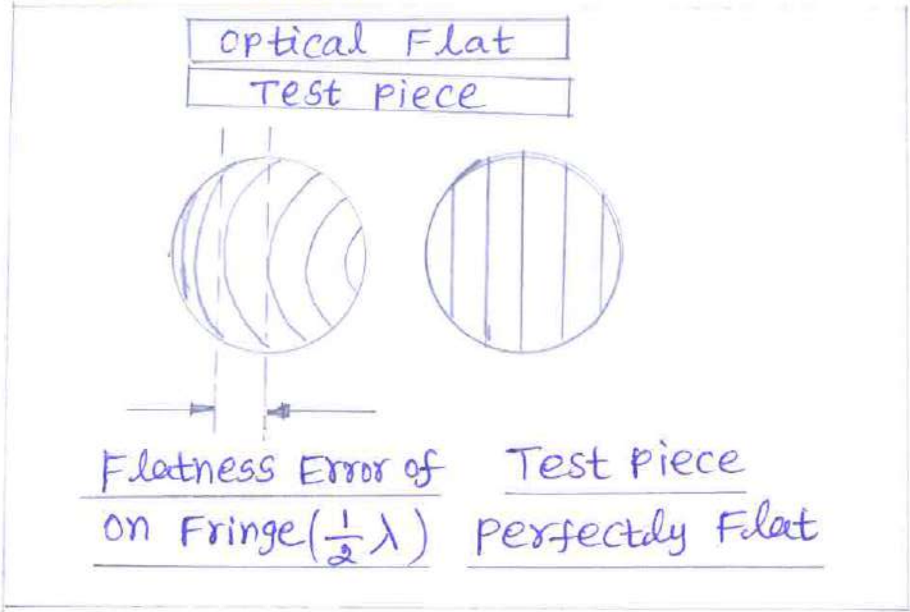
Optical Flat

* An optical flat is an optical-grade piece of glass lapped and polished to be extremely flat on one or both sides, usually within a few millionths of an inch (about 25 nm).
* They are used with a monochromatic light to determine the flatness of other optical surfaces by interference.
* When an optical flat is placed on another surface and illuminated, the light waves reflect off both the bottom surface of the flat and the surface is resting on.

* This causes a phenomenon similar to thin-film interference.
* The reflected waves interfere, creating a pattern of interference fringes visible as light and dark bands.




* The spacing between the fringes is smaller where the gap is changing more rapidly, indicating a departure from flatness in one of the two surfaces, in a similar way to the contour lines on a map.
* A flat surface is indicated by a pattern of straight, parallel fringes with equal spacing, while other patterns indicate uneven surfaces.
* Two adjacent fringes indicate a difference in elevation of one-half wavelength of the light used, counting the fringes differences in elevation of the surface can be measured to millionths of an inch.

* Usually only one of the two surfaces of an optical flat is made flat to the specified tolerance and this surface is indicated by an arrow on the edge of the glass.

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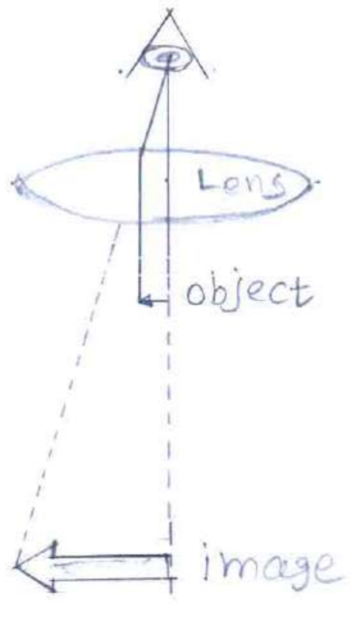
## Optical Microscope

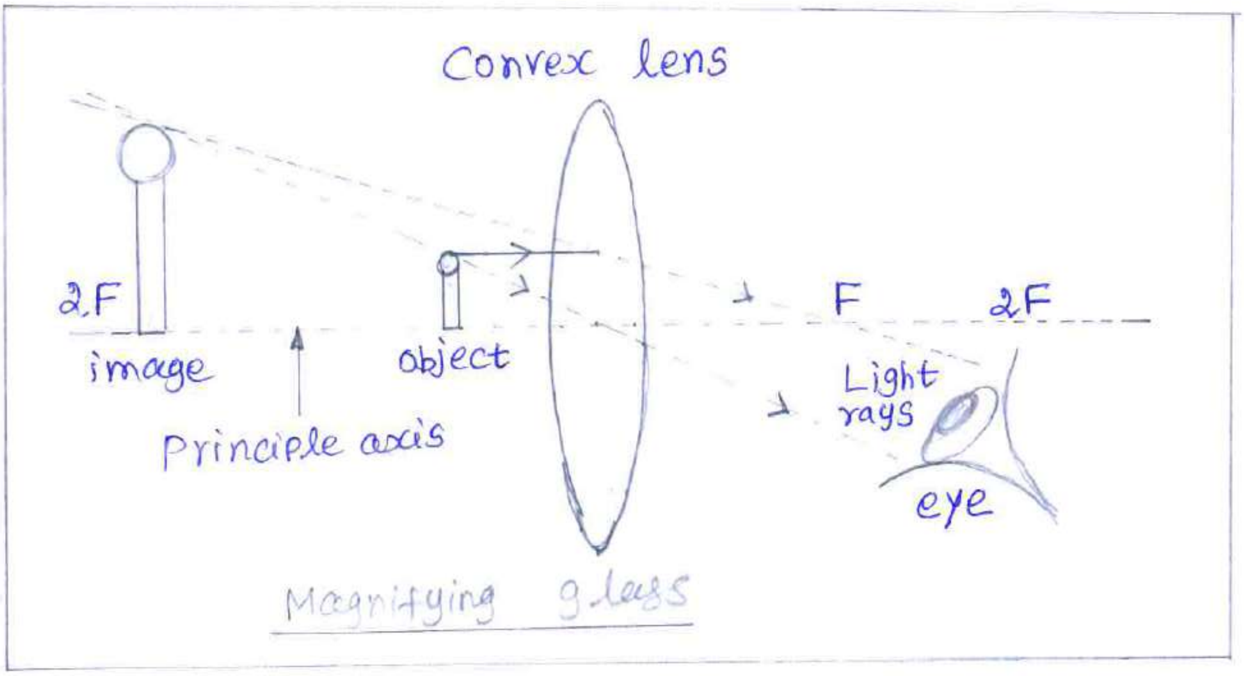
* The optical microscope is a type of microscope which uses visible light and a system of lenses to magnify images of small samples.
* optical microscopes are the oldest design of microscope and invented in 17th century.
* Basic optical microscopes can be very simple, although there are many complex designs which aim to improve resolution and sample contrast.
* The image from an optical microscope can be captured by normal light-sensitive cameras to generate a micrograph.



* original images were captured by photographic film. charge coupled device (CCD) Cameras allow the capture of digital images.
* Purely digital microscopes are now available which use a CCD Camera to examine a sample, showing the resulting image directly on a Computer screen without the need for eyepieces.

Magnifying Glass:

* Magnifying glass is a convex lens that is used to produce a magnified image of an object.
* The lens is usually mounted in a frame with a handle.
* A magnifier consists of many very narrow concentric ring-shaped lenses, such that the combination acts as a single lens but thinner. This arrangement is Known as Fresnel lens.
* A magnifying glass can be used to focus light such as to concentrate the sun's radiation to create a hot spot at the focus for fire starting.




* The magnification of a magnifying glass depends upon where it is placed between the user's eye and the object being viewed, and the total distance between them.
* The magnifying power is equivalent to angular magnification. The magnifying power is the ratio of the sizes of the images formed on the user's retina with and without the lens.

## Applications of visual inspection

* Inspection of corrosion, erosion, cracks, fracture etc
* Identification of any leakage, abnormal operations etc.
* Finding of misalignment of parts in the equipments
* Inspection of weld defects such as surface cracks lack of penetration, tear cracks, excess reinforcement porosities, mismatch etc.,
* Identification of minute discontinuities with the help of optical aids in pumps, compressors, turbo generator parts, instruments etc.

# UNIT-4 MATERIAL CHARACTERIZATION TESTING

* Material characterization is the process of measuring and determining physical, chemical, mechanical and microstructural properties of materials.
* There are two types
* microscopy testing
* Macroscopic testing

## Microscopy

* Microscopy is a technique that allows the determination of both the composition and the structure of a material.
* It is essentially the process of viewing the structure on a much finer scale not possible with the naked eye.
* The properties of materials with extremely fine features and defects those are only possible to observe using microscopic techniques.

## Microscopic properties of materials

* contaminants & purity
* Ingredients
* chemical bonding

* Molecular pattern
* crystal structure & lattice bonding
* Nano size

Microscopic Instruments

* Optical microscope
* Scanning Electron Microscope
* Transmission Electron Microscope
* Field Ion Microscope
* Atomic Force Microscope
* X-ray diffraction topography

Macroscopic observations

* In which some physical and chemical changes are observed.
* This change can be observed by the naked eye.
* This simple process can yield a large amount of information about the material, such as the colour of the material, its luster, its shape, its composition, its structural features, etc.

## Macroscopic properties of Materials

* Density
* Volume
* Strength
* Hardness
* Roughness, etc.,

## Macroscopic Instruments

* Mechanical testing, including tensile, compressive, torsional, creep, fatigue, toughness and hardness testing.
* Differential thermal analysis
* Dielectric thermal analysis
* Thermogravimetric analysis
* Differential Scanning Calorimetry
* Ultrasonic techniques

## Optical Microscope

* The optical microscope, also referred to as a light microscope that commonly uses visible light and a system of lenses to generate magnified images of small objects.

## Principle

* The functioning of the light microscope is based on its ability to focus a beam of light through a specimen, which is very small and transparent, to produce an image.
* The image is then passed through one or two lenses for magnification for viewing.
* The transparency of the specimen allows easy and quick penetration of light.

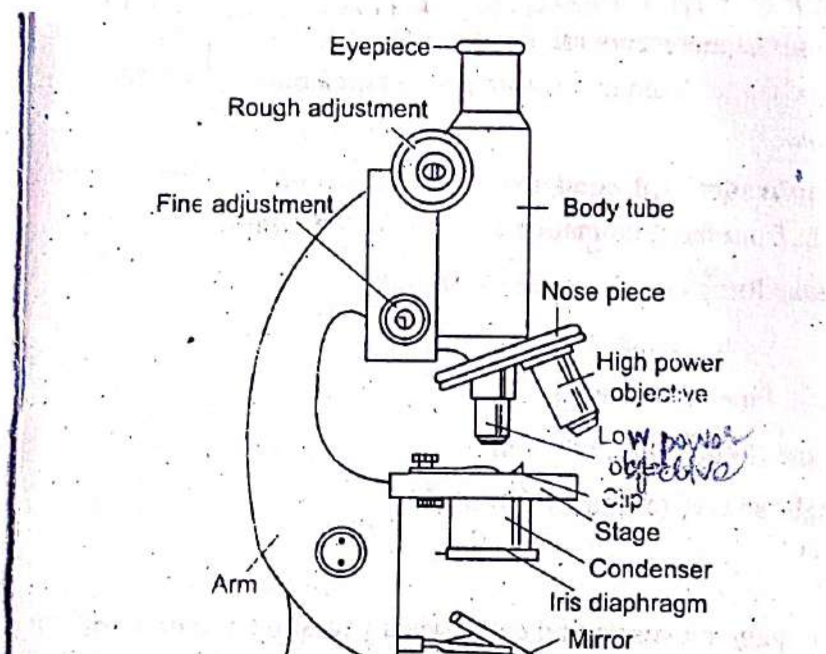



*Fig. 4.2. Typical cross section of optical microscope*

## Components

* Eye piece
* Objective turret
* Objective lenses
* Diaphragm and condenser
* Focus Knobs
* Stage
* Light source

## Working

* The stage moves up and down when you turn a thumb wheel on the side of the microscope.
* By raising and lowering the stage, you move the lenses closer to or further away from the object you are examining, adjusting the focus of the image.
* The slide is held in place by two metal clips, one on either side.
* Light traveling up from the mirror passes through the glass slide, specimen and cover slip to the objective lens.

* This makes the first magnification; its work by spreading out light rays from the specimen so they appear to come from a bigger object.
* The objective lens usually consists of more than one lens.
* A selection of other objective lenses can be used to magnify the specimen by more or less.
* The eyepiece lens maginifies the image from the objective lens, rather like a magnifying glass.
* Some microscopes, you can move the eyepiece up and down by turning a wheel. This gives you fine control of the focus.

Magnification

* The maximum magnification power of optical microscopes is typically limited to around 1000X because of the limited resolving power of visible light.

## Advantages

* It is relatively easy to use.
* It is small and lightweight.
* It offers high levels of observational quality.
* It is unaffected by electromagnetic fields.
* It does not require radiation to operate.
* It requires very little training.
* Minor maintenance cost
* It is fully adjustable.

## Disadvantages

* Low magnification
* Separate Sample Preparation
* Poor surface view.
* Light microscopes cannot operate in darkness
* Light microscopes cannot provide three-dimensional renderings.

## Applications

* Optical microscopy is used extensively in microelectronics, nanophysics, biotechnology, research, mineralogy and microbiology.

* optical microscopy is used for medical diagnosis.
* In industrial use, binocular microscopes are common.
* In certain applications, long-focus microscopes are beneficial.

## Electron microscopy

* An electron microscope is a microscope that uses a beam of accelerated electrons as a source of illumination.
* As the wavelength of an electron can be up to 100,000 times shorter than that of visible light photons, electron microscopes have a higher resolving power than light microscopes and can reveal the structure of smaller objects.

## Types of Electron microscope

* Transmission Electron Microscope
* Scanning Electron microscope

# Scanning Electron Microscope (SEM)

* A scanning electron microscope (SEM) uses a focused electro probe to extract structural and chemical information point on the specimen.
* It use wide range of scale from nanometer to micrometer.

## Principle

* A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons.
* The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample.

## Components:

* Electron gun
* condenser lens
* Vacuum chamber

* Deflector coils
* Secondary electron detector
* Image display and Recording
* Specimen stage

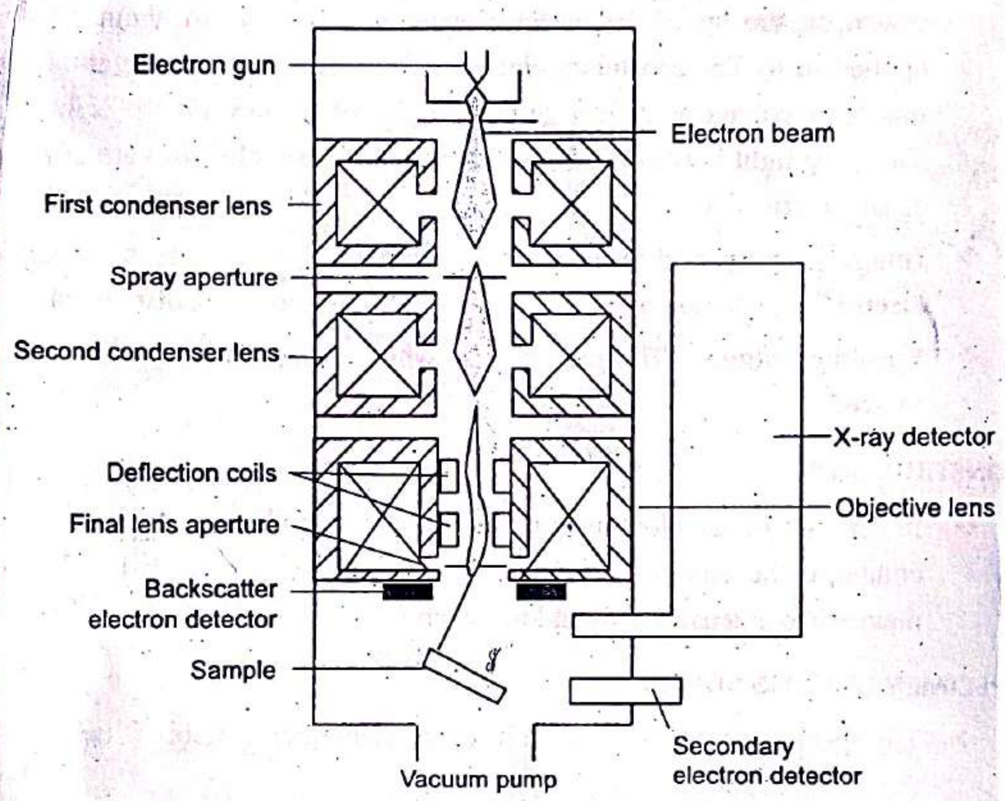



*Fig. 4.4. Sectional view of SEM*

## Working

* In SEM an electron beam is emitted from an electron gun fitted with a tungsten filament cathode.
* The electron beam, which typically has an energy ranging from 0.2 KeV to 40 KeV, is focused by condenser lenses.

* The beam passes through pairs of scanning coils or pairs of deflector plates in the electron column.
* When the primary electron beam interacts with the sample, the electrons lose energy by repeated random scattering and absorption.
* The energy exchange between the electron beam and the sample results in the reflection of high-energy electrons by elastic scattering, emission of secondary electrons, backscattered electrons and characteristic X-rays by inelastic scattering and the emission of electromagnetic radiation, each of which can be detected by specialized detectors.
* The beam current absorbed by the specimen can also be detected and used to create images of the distribution of specimen current.
* Electronic amplifiers of various types are used to amplify the signals, which are displayed as variations in brightness on a computer monitor.

## Magnification

* Magnification in an SEM can be controlled over a range of about 6X orders of magnitude from about 10 to 3,000,000 times.
* Magnification is therefore controlled by the current supplied to the scanning coils or the voltage supplied to the deflector plates and not by objective lens power.

## Advantages

* Rapid, high-resolution imaging
* Quick identification of elements present
* Excellent depth of field
* Versatile platform that supports many other analysis techniques
* Low vacuum mode enables imaging of insulating and hydrated samples.

## Limitations

* Size restrictions may require cutting the sample.
* The size is not portable
* SEMs are expensive and large
* Maintenance involves keeping a steady voltage, currents to electromagnetic coils and circulation of cool water.

## Applications of SEM

* Detailed surface topography images
* Failure analysis
* Dimensional analysis
* Process characterization
* Reverse Engineering
* Particle identification
* Surface 3D
* Elemental analysis

## Transmission Electron Microscopy (TEM)

* A Transmission Electron Microscope (TEM) utilizes energetic electrons to provide morphologic, compositional and crystallographic information on samples.
* The transmitted electrons that have passed through the thin sample are detected to form images, which is the reason to call it transmission electron microscopy.
* Magnification of 1 nanometer TEMs are the most powerful microscopes.

## Principle

* An image is formed from the interaction of the electrons with the sample as the beam is transmitted through the specimen.
* The image is then magnified and focused onto an imaging device, such as a fluorescent screen, a layer of photographic film, or a sensor such as a scintillator attached to a charge-coupled device.

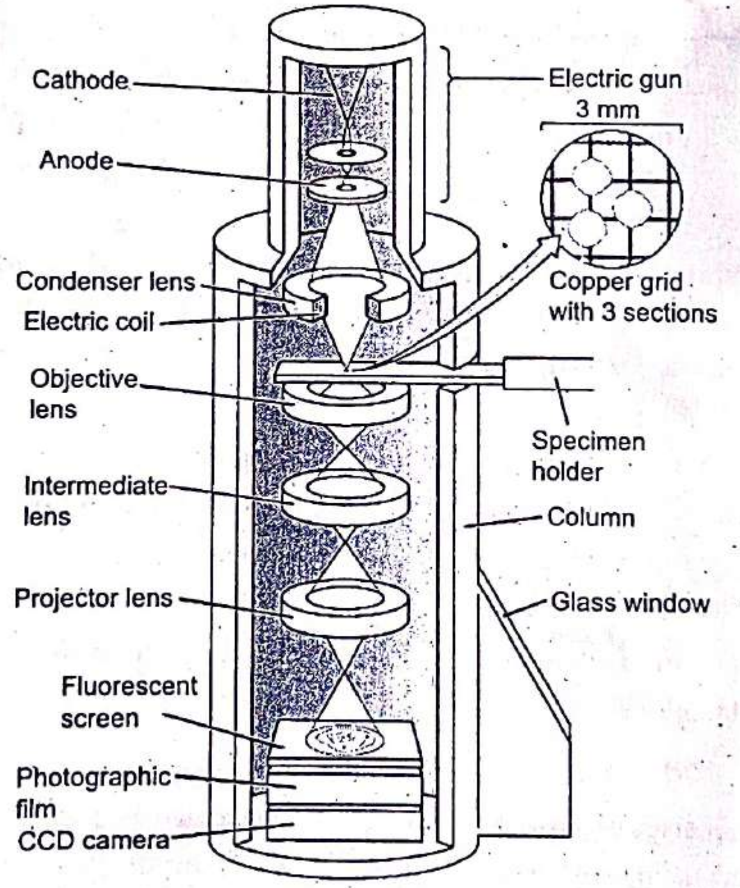



*Fig. 4.6. Working of TEM*

## Components of TEM

* Electron source
* Electromagnetic lenses
* Vacuum chamber
* condensers
* Sample stage
* phosphor or fluorescent screen
* condenser lens
* objective aperture

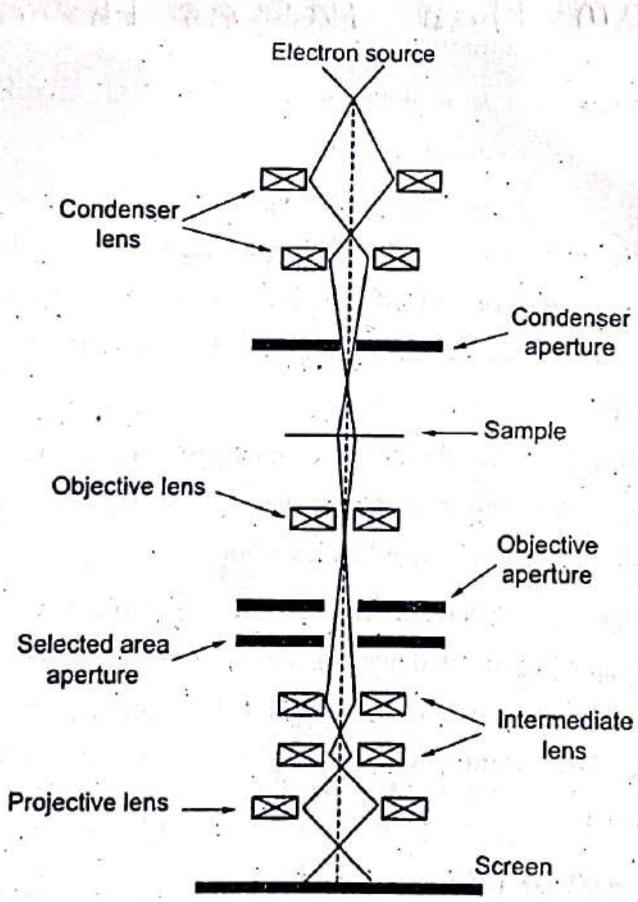



*Fig. 4.7. Cross sectional view of TEM*

## working

* TEMs employ a high voltage electron beam in order to create an image.
* An electron gun at the top of a TEM emits electrons that travel through the microscope's vacuum tube.
* Rather than having a glass lens focusing the light, it employs an electromagnetic lens which focuses the electrons into a very fine beam.
* This beam then passes through the specimen, which is very thin, sample thickness is less than 200 nm, depending on the composition of sample and the expected information from TEM characterization.
* The electrons either scatter or hit a fluorescent screen at the bottom of the microscope.
* During transmission, the speed of electrons directly correlates to electron wavelength; the faster electrons move, the shorter wavelength and the greater quality and detail of the image.

* An image of the specimen with its assorted parts shown in different shades according to its density appears on the screen.
* The image becomes visible when the electron beam hits a fluorescent screen at the base of the machine.

Advantages

* The highest spatial resolution elemental mapping of any analytical technique.
* Small area crystallographic information.
* Strong contrast between crystalline vs amorphous materials without chemical staining.
* TEMs offer the most powerful magnification, potentially over one million times or more.
* It can be utilized in a variety of different scientific, educational and industrial fields.
* Images are high quality and detailed
* TEMs are able to yield information of surface features, shape, size and structure.

## Disadvantages

* Significant sample preparation time
* Small sampling volumes and samples are typically 100 nm thick.
* TEMs are large and very expensive.
* Operation and analysis requires special training
* Images are black and white
* Electron microscopes are sensitive to vibration and electromagnetic fields.

## Applications

* Metrology at 0.2 nm resolution
* Determination of crystallographic phases at the nanometer scale.
* Catalyst studies
* Nanometer scale elemental maps
* Super lattice characterization
* Crystal defect characterization.
* Chemical information such as composition and bonding
* Energy filtered imaging.
* It can be used in semiconductor analysis
* Colleges and Universities can utilize TEMs for research and studies.

Comparison between SEM and TEM 

| Category | SEM | TEM |
|---|---|---|
| Source electrons | scattered electrons | Transmitted electrons |
| Process of working | Scattered absorption | Diffraction |
| Energy | 1-30 KV | 60-300 KV |
| Environment | Air/Vacuum | Vacuum |
| Specimen thickness | Any thickness | less than 150 nm |
| Output | 3D image formation | 2D projection image of inner structure |
| Property identification | Roughness or Contamination detection | Structural defects or impurities |
| Magnification | 2 million level magnification | 50 million level magnification |
| Field of view | Large | Limited |
| Optimum resolution | 0.4 nm resolution | 0.5 angstroms resolution |
| Image formation | Electron are captured and countered by detector image on PC | Direct image on fluorescent screen or PC screen with LCD |
| Operation | Little sample preparation | Laboratory sample preparation |

# Diffraction Techniques

## Diffraction

* Diffraction refers to various phenomena that occur when a wave encounters an obstacle or a slit.
* It is defined as the bending of waves around the corners of an obstacle or through an aperture into the region of geometrical shadow of the obstacle.

## Diffraction principle

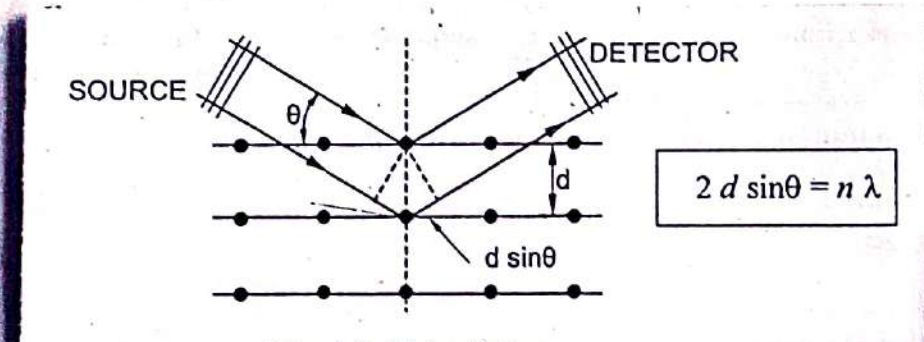



*Fig. 4.8. Bragg's law*

* Bragg's law is which determines the angles of coherent and incoherent scattering from a crystal lattice.
* when X-rays are incident on a particular atom, they make an electronic cloud move just like an electromagnetic wave.

* There are two conditions for constructive interference of waves:

1. The angle of incidence must equal the angle of reflection
2. The difference in path length must be an integral number of wavelengths.

Methods of diffraction

* Electron diffraction
* Neutron diffraction
* X-ray diffraction

Advantages

* Data generation is quick.
* Testing is cheap but equipment installation is costlier.

Disadvantages

* Sample preparation is complex
* Absorption of radiation
* Source is costlier
* Most of the diffraction method need vacuum.

## Applications

* Diffraction methods offer a unique way to measure micro stresses in crystalline materials, because each phase will have its own diffraction pattern giving information on the stresses in that phase.
* It is also the measurement of changes in crystal plane spacing in different directions with respect to the specimen surface.

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## Spectroscopic Techniques

### Spectroscopy

* Spectroscopy deals with the production, measurement and interpretation of spectra arising from the interaction of electromagnetic radiation with matter.
* Spectroscopic methods are very informative and widely used for both quantitative and qualitative analysis.

## principle of spectroscopy

* The beam of electromagnetic radiation onto a sample and observe how it responds to such a stimulus.
* The response is usually recorded as a function of radiation wavelength.
* A plot of the response as a function of wavelength is referred to as a spectrum.
* The Beer - Lambert law states that the quantity of light absorbed by a substance dissolved in a fully transmitting solvent is directly proportional to the concentration of the substance and the path length of the light through the solution.

## Methods of spectroscopy

* The method of spectroscopy differ with respect to the species to be analyzed, the type of radiation - matter interaction to be monitored and the region of the electromagnetic spectrum used in the analysis.

* spectroscopic methods based on the absorption or emission of radiation in the ultraviolet, visible, infrared and radio.
* Each of these methods is distinct in that it monitors and different types of molecular or atomic transitions.
* A distinct feature of electromagnetic radiation is mentioned in the Table.

| Name of Spectroscopy | Type of radiation used | wavelength | Relative Energy | what it does to the molecule/atom | what it tells us about the atom/molecule |
|---|---|---|---|---|---|
| Photo electron Spectroscopy | X-rays | 0.01 to 10 nm | Very high | Remove core electrons | In Atomic structure, it gives information about how tightly the electrons are held by the nucleus |
| UV-visible Spectroscopy | ultra violet | 50 to 400 nm | High | Excites valence electrons | Identify the molecular element |
| UV-visible Spectroscopy | Visible light | 400 to 800 nm | Medium | Excites Valence electrons | Concentration of a molecule |
| IR Spectroscopy | Infra red | 2.5 to 50 $\mu m$ | Low | changes the vibrations in covalent bonds | Types of bonds/atoms within a molecule |
| microwave Spectroscopy | Micro wave | 0.3 mm to 0.5 m | Very Low | changes the rotations of the atoms in covalent bonds | Location of hydrogen atoms within a molecule |

## Advantages of spectroscopy

* cure monitoring of composites using optical fibers.
* Measurement of different compounds in food samples by absorption Spectroscopy both in visible and infrared spectrum.
* Measurement of toxic compounds in blood samples.
* Non-destructive elemental analysis
* Electronic structure research with various spectroscopes.
* Quantitative and qualitative analysis

## Disadvantages of spectroscopy

* Radiation may be easily Contaminated.
* cost of spectroscopy equipment is high.
* Not suitable for all Kind of material.
* Need low working temperature at certain condition.

## Applications

* Understanding constitution of matter from atoms to complex molecules
* Studies on diverse materials existing in nature from deep sea studies to space missions.

* Investigations of crime samples.
* Analysis and development of whole range of man-made materials.
* Studies on environmental samples
* Mineralogy

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## Atomic spectroscopy or Flame spectroscopy

* Atomic spectroscopy is based upon the absorption or emission of electromagnetic radiation by atomic particles.
* Spectroscopic determination of atomic species can be performed on a gaseous medium in which the individual atoms or elemental ions.
* Energy transitions of outer electrons of atoms after volatilization in a flame.
* Liquid solution samples are aspirated into a burner or nebulizer, desolvated, atomized and sometimes excited to a higher energy electronic state.
* Atomic spectroscopy is the study of the electromagnetic radiation absorbed and emitted by atoms.

## principle

* The electons of the atoms in the atomizer can be promoted to higher orbitals for a short amount of time by absorbing a set of energy.
* This amount of energy is specific to a particular electron transition in a particular element, and in general, each wavelength corresponds to only one element.

## Components

* Lamp source
* Nebulizer
* Atomizer
* Monochromator
* Detector

## Working

* A sample is volatilized and decomposed to produce gas phase atoms and ions.
* Atomization is a critical step in all atomic spectroscopy.
* Flames and electro thermal atomizers are widely used in atomic absorption spectrometry.

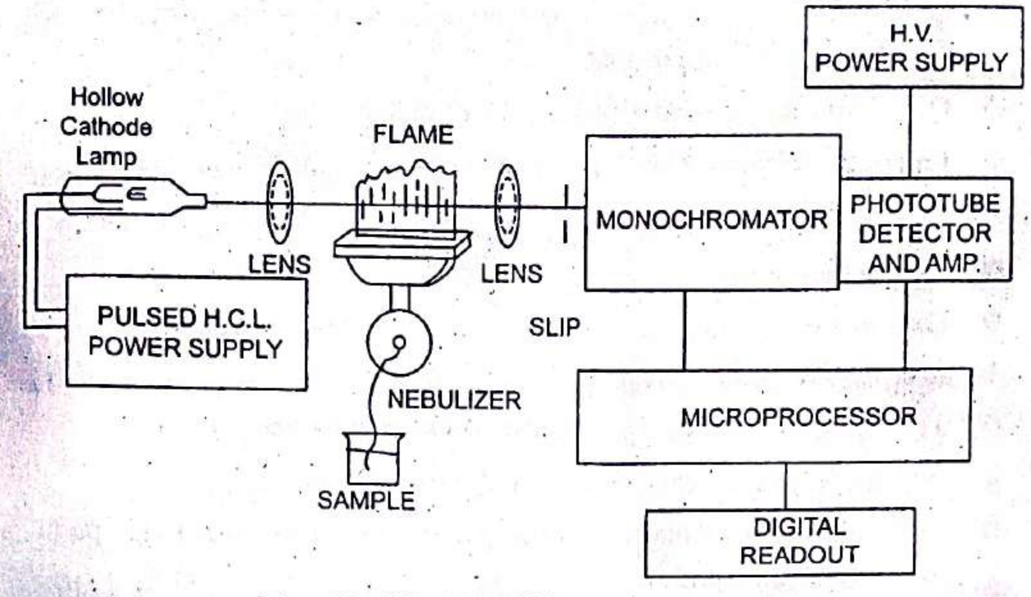



*Fig. 4.9. Working of flame spectroscopy*

* In the components of an atomic absorption or flame absorption apparatus, the flame can be considered to be a dilute gaseous solution of the atomized sample.
* Radiation of specific wavelength is emitted by the hollow cathode lamp onto the gaseous atoms in the atomizer.
* The monochromator focuses the specific wavelengths onto the detector.
* The detector finds the amount of light absorbed.
* The concentration of atoms in the sample is directly proportional to the absorbance.

## Advantages

* High sensitivity
* Easy to use
* In expensive
* The method of analysis is very simple and economical.
* It is quick, convenient, selective and sensitive analysis.

## Disadvantages

* Different cathode lamp for different elements
* can detect only metals and some of the non-metals
* only one element is detected
* The elements such as carbon, hydrogen, and halides cannot be detected due to their non-radiating nature.
* only liquid samples may be used.

## Applications

* Level of metal could be detected in tissue samples like aluminium in blood and copper in brain tissues.
* Presence of metals as an impurity or in alloys could be found easily.

* Determination of elements in the agricultural and food products.
* Determination of lead in petrol.
* Determination of calcium and magnesium in cement.
* Determination of the elements present in different samples like food, water, waste water, nano material, biomaterials and industrial waste.

## UV / visible spectroscopy

* UV spectroscopy is based upon absorption of electromagnetic radiation in the visible and ultraviolet regions of the spectrum resulting in changes in the electronic structure of ions and molecules.
* The wavelength of UV and visible light are substantially shorter than the wavelength of infrared radiation.
* The UV spectrum ranges from 200 to 700 nm.
* when a molecule or ion absorbs ultraviolet or visible radiation it undergoes a change in its valence electron transition.

## Principle

* Diminution of a beam of light after it passes through a sample or after reflection from a sample surface.
* Absorption measurements can be at a single wavelength or over an extended spectral range.
* Energy transitions of bonding and non-bonding outer electrons and molecules, usually delocalized electrons.

## Components

* Light source
* Monochromator
* Detector
* Recording devices

## Working

* Polychromatic light from the source is focused on the entrance slit of a monochromator, which selectively transmits a narrow band of light.
* This light then passes through the sample area to the detector.

* The absorbance of a Sample is determined by measuring the intensity of light reaching the detector without the sample and comparing it with the intensity of light reaching the detector after passing through the sample.

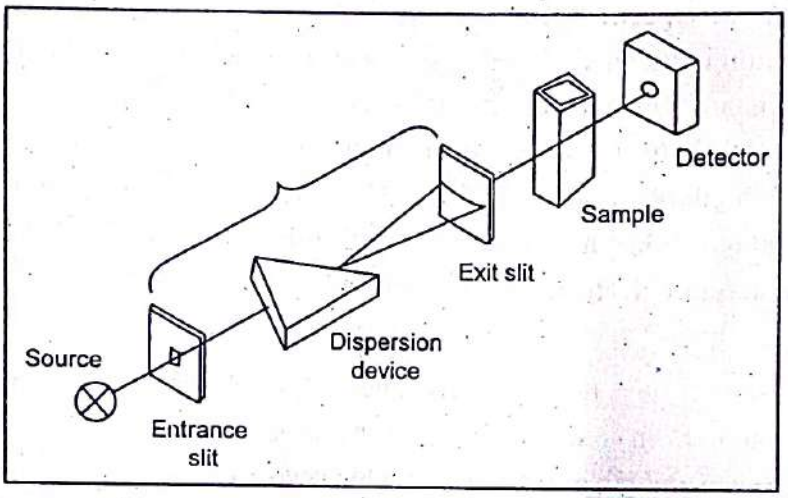



*Fig. 4.10. Working of UV spectroscopy*

## Advantages

* Minimum damage to sample.
* Better result at lower concentration
* Very rapid Calibration
* High Sensitivity
* Good accuracy

## Disadvantages

* Lack of Sensitivity
* Instrument is expensive

* Have limited application to identify the functional group or particular molecule as a result of absorption spectra.

Applications

* Routine qualitative and quantitative measurement
* Used to find relative purity of a solution.
* Widely applicable to both organic and inorganic compounds.

---

Electrical Techniques

* Electrical properties are a key physical property of conducting materials.
* It is often necessary to accurately measure the resistivity of materials.

Principle

* An electrochemical cell is used to house the chemical reaction and is electrically connected to the electrochemical spectrometer to obtain the electrical response of an electrolytic solution.
* Electrochemical Impedance Spectroscopy (EIS) systems are operated using computer programs specifically designed for EIS testing.

* Therefore, prior to conducting an EIS experiment is essential that all components of the system be attained.

## Components

* Three electrodes
* Electrolytic solution
* Insulating material
* Display unit

## Working

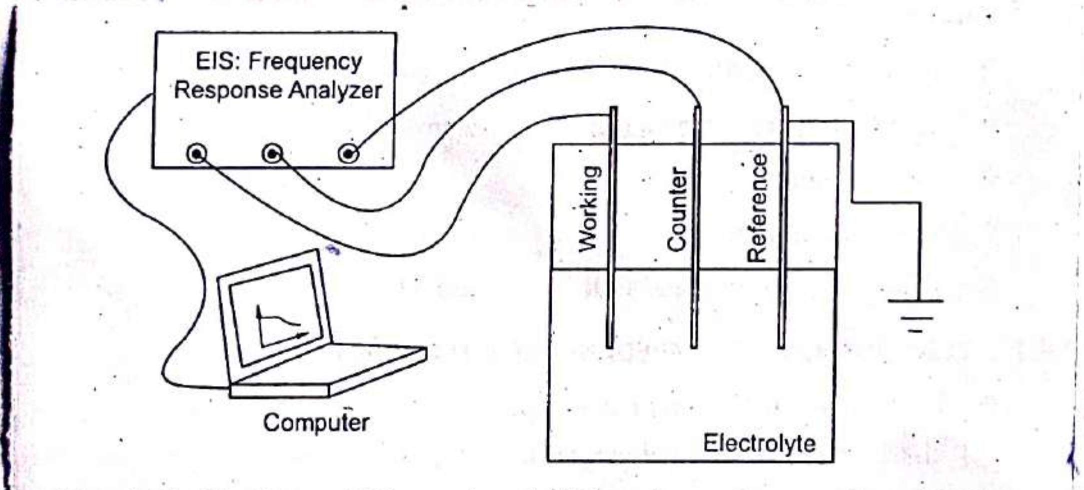



*Fig. 4.11. Working of Electrochemical Impedance Spectroscopy (EIS)*

* EIS studies utilize a three electrode mode which is comprised of a working electrode, a counter electrode and a reference electrode.
* While electrode geometries may vary the general experimental setup remains similar to the procedure outlined below.

* The three electrodes are mounted on an electrode stage and secured.
* The electrolytic solution is prepared and transferred to the sample container.
* A metallic sample container would provide additional pathways for electrons during experimentation leading to a reduction in the EIS current response as electrons move into the metal rather than the reference electrodes.
* The sample container should be composed of an insulating material, such as glass or plastic, which will not interfere with the transfer of electrons during testing.
* The electrode mount is then placed on the sample container such that a portion of each electrode is submerged in the electrolytic solution.
* Four leads are used to attach the three electrodes to the EIS frequency response analyzer.

* A working lead and a counter lead is used to carry current, whereas the working sense lead and reference leads are used to sense voltage.
* The working sense lead connects the exposed end of the electrode to the EIS.
* The reference lead is attached to the reference electrode and the counter lead is connected to the counter electrode.
* The fourth lead is recommended to ground the system during testing.
* once all leads are connected and by stimulus the data is collected from computer generated data.
* The impedance produced during electro-chemical experimentation can be evaluated through use of one or more equivalent circuits.

Advantages

* useful on high resistance materials such as paints and coatings.
* Time dependent data is available
* Non-destructive

* Quantitative data available
* Use service environments

## Disadvantages

* Expensive
* Complex data analysis for quantification

## Applications

* It provides information about the corrosion kinetics and coatings evaluation.
* It is an accurate and reproducible technique suitable for highly resistive environments.
* It provides data about the electrochemical control mechanism, indicating if corrosion occurs by activation, concentration or diffusion.
* It characterizes the state of the rebar and the morphology of the corrosion.
* It allows for monitoring of the evolution of the passive or active state over time.

## Magnetic Techniques

* Magnetic methods are potential methods for evaluation of surface manifestations such as microstructural degradation, residual stresses, surface roughness and defect detection in surface coatings of magnetic substrates.
* Electromagnetic methods have very high potential for material characterization and well known nondestructive testing.
* Electromagnetic techniques are able to indicate non-destructively and quickly changes of residual stresses, texture, micro structure states and mechanical properties.

## Principle:

* Magnetic hysteresis occurs when an external magnetic field is applied to a ferromagnetic such as iron and the atomic dipoles align themselves with it.

* when the field is removed, part of the alignment will be retained: the material has become magnetized.

Advantages

* Non destructive technique
* Used for nano material characterization
* Immediate result
* Accuracy in measurement of dielectric losses.

Disadvantages

* Limitation of lateral resolution.
* Need very thin samples
* characterization limited to dielectric permittivity.
* Multiple steps
* Need technical knowledge
* Presence of air gaps may reduce the accuracy.

Applications

* Used to find microstructure, texture, hardness depth, Phase content, residual stress, aging and grain size.

# UNIT 5 OTHER TESTING

## Thermal testing

* Thermal testing involves testing a product at the extremes of its intended use thermal environment for heating rate, temperature and airflow or gaseous atmosphere or vaccum with measuring case temperatures on individual components to determine the effect on product performance and long-term reliability.
* It measures based on dynamic relationships between temperature, Mass, volume and heat of reaction.

Parameters of thermal testing:-

| Method | Parameter testing |
| --- | --- |
| Thermogravimetric analysis | Mass changes |
| Differential thermal analysis | Temperature difference |
| Differential Scanning Calorimetry | Heat difference |
| Evolved gas analysis | Gas decomposition |
| Thermo mechanical analysis | Deformation and dimension |
| Dilatometer | Volume |
| Dielectric thermal analysis | Electrical Properties |
| Thermo optical analysis | optical Properties |

## Thermogravimetric Analysis (TGA)

* Thermogravimetric analysis (TGA) is a type of thermo analytical testing performed on materials to determine changes in weight in relation to changes in temperature.
* TGA relies on a high degree of precision in three measurements: weight, temperature and temperature change.
* TGA is commonly employed in research and testing to determine characteristics of materials, to determine degradation temperatures, absorbed moisture content of materials, the level of inorganic and organic components in materials, decomposition points of explosives and solvent residues.

## Differential Scanning Calorimetry (DSC)

* DSC measures the energy absorbed or released from a sample as a function of time or a temperature profile.
* DSC is useful to make the measurements for melting points, heats of reaction, glass transition and heat capacity.

## Principle

* Differential scanning calorimetry (DSC) is based on the principle, sample and reference are maintained at the same temperature, even during a thermal event.
* The energy required maintaining zero temperature different between the sample and the reference is measured.
* By calibrating the standard material, the unknown sample quantitative measurement is achievable.

## Power compensation DSC

* A technique in which difference of thermal energy that is applied to the sample and the reference material separately per unit of time is measured as a function of the temperature.

## Components

* Sample holder
* Sensors
* Furnace
* Temperature controller

Working:

* The power needed to maintain the sample temperature equal to the reference temperature is measured.

* In power compensation DSC two independent heating units are employed.

* These heating units are quite small, allowing for rapid rates of heating, cooling and equilibration.

* The heating units are embedded in a large temperature controlled heat sink.

* The sample and reference holders have platinum resistance thermometers to continuously monitor the temperature of the materials.

* The instruments records the power difference needed to maintain the sample and reference at the same temperature as a function of the programmed temperatures.

* Power compensated DSC has lower sensitivity than heat flux DSC, but its response time is more rapid.

* It is also capable of higher resolution than heat flux DSC.

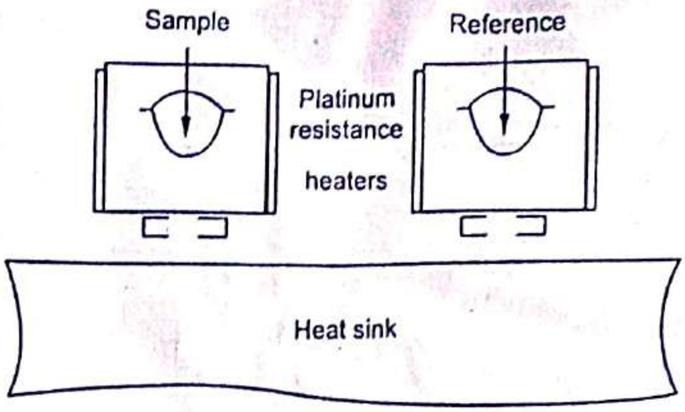



*Fig. 5.1. Typical arrangement of Power compensated DSC*

* This makes power compensated DSC well suited for kinetics studies in which fast equilibrations to new temperature settings are needed.

Heat flux DSC

* In heat flux DSC, the difference in heat flow into the sample and reference is measured while the sample temperature is changed at the constant rate.

* Sample and reference are connected by a low resistance heat flow path.

* The assembly is enclosed in a single furnace.

## components

* Sample holder
* Sensors

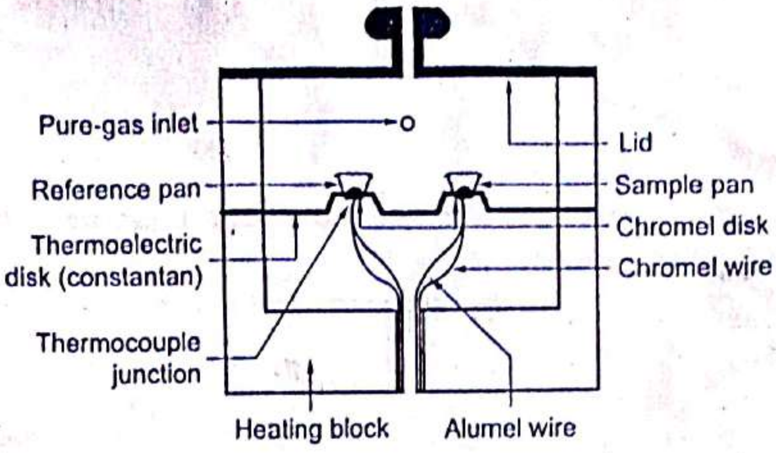



*Fig. 5.2. Typical arrangement of heat flux DSC*

## working

* The main assembly of the DSC cell is enclosed in a cylindrical, silver heating black, which dissipates heat to the specimens via a constantan disc which is attached to the silver block.
* The disc has two raised platforms on which the sample and reference pans are placed.
* A chromel disc and connecting wire are attached to the underside of each platform, and the resulting chromel - constantan thermocouples are used to determine the differential temperatures of interest.

* Alumel wires attached to the chrome discs provide the chromel-alumel junctions for independently measuring the sample and reference temperature.

## DSC curve

* DSC curve is plot between heat flow and temperature. It shows various peaks of measurement.

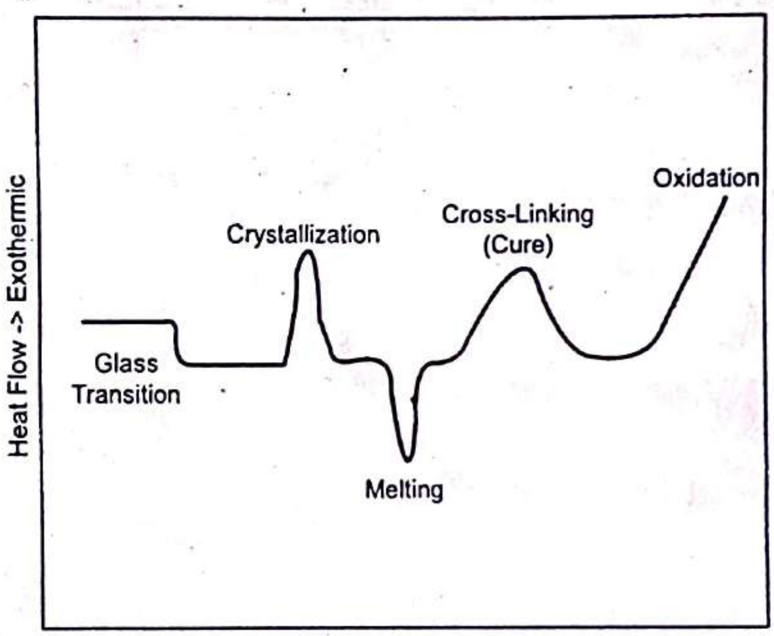



*Fig. 5.3. DSC Curve*

## Advantages of DSC

* Instruments can be used at very high temperatures.
* Instruments are highly sensitive
* Flexibility in sample volume
* Characteristic transition or reaction temperatures can be determined.

* High resolution obtained.
* High sensitivity
* Stability of the material

Limitations of DSC

* DSC generally unsuitable for two-phase mixtures
* Difficulties in test cell preparation in avoiding evaporation of volatile solvents.
* DSC is generally only used for thermal screening of isolated intermediates and products.
* Does not detect gas generation
* Uncertainity of heats of fusion and transition temperatures

Differential Thermal Analysis (DTA)

* Differential thermal analysis (DTA) is a thermo-analytical technique which is used for thermal analysis where thermal changes can be studied.
* It is used to determine the oxidation process, decomposition and loss of water or solvent.

## Principle:

* In DTA, the sample material and an reference material are made to undergo identical thermal cycles, while recording any temperature difference between Sample and reference.
* This differential temperature is then plotted against time, or against temperature
* Changes in the sample, either exothermic or endothermic can be detected relative to the inert reference.

## Components

* Furnace
* Sample holder
* DC amplifier
* Differential temperature Detector
* Furnace temperature Program
* Recorder
* Control Equipment

## Working

* The sample under investigation is loaded into a container.
* This container is then placed onto the sample pan and it is marked as S. (S means Sample)

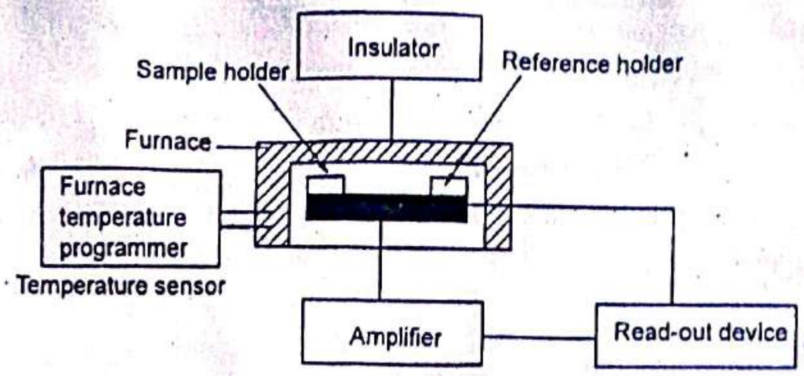



*Fig. 5.4. Cross section of DTA*

* Same quantity of reference sample is placed in another container which is then placed onto the reference pan and it is marked as R (R means reference)
* In order to heat the sample pan and the reference pan at an identical rate, the dimensions of these two pans should be nearly identical.
* Moreover, the sample and the reference should have equal weights, thermally matched and should be arranged symmetrically with the furnace.
* The metal block which surrounds the pans acts as a heat sink whose temperature is increased slowly by using an internal heater.
* The sink then heats the sample and reference material simultaneously.

* Two pairs of thermocouples are used, one pair is in contact with the sample and the second pair is in contact with the reference.
* Thermocouple is attached with an amplifier which amplifies the result of differential thermocouple and sent this result to the read-out device which displays the result in the form of DTA curve or thermogram as a function of the sample temperature, reference temperature or time.
* No signal is generated if no temperature difference is observed even though the actual temperatures of both the sample and reference are increasing.
* when there is a physical change in the sample then heat is absorbed or released.
* This is an endothermic reaction where the heat is absorbed and the temperature of the sample is decreased.
* Now the sample is at a lower temperature than that of the reference.
* This temperature difference between sample and reference produces a net signal, which is then recorded.

## DTA curve

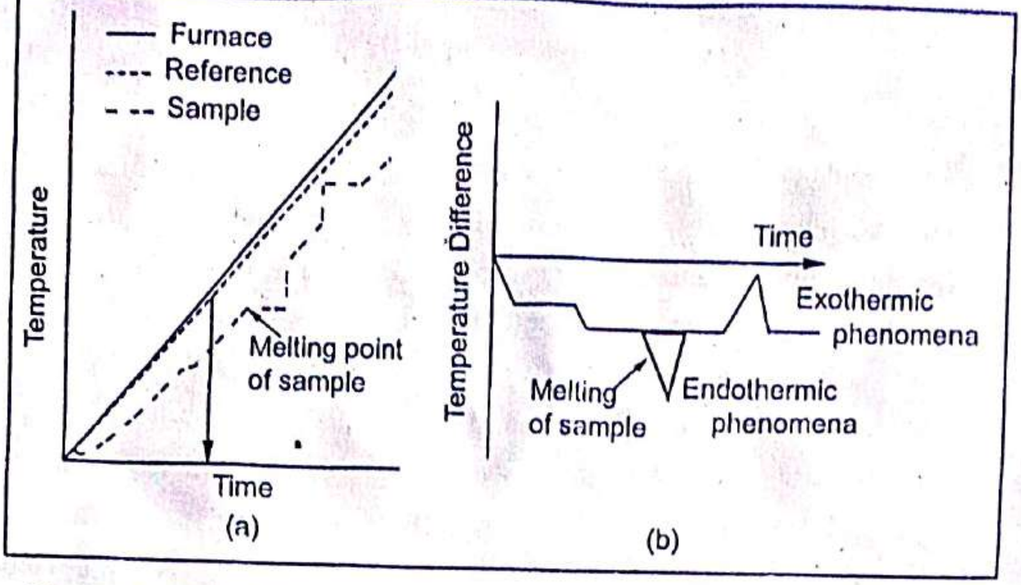



***Fig. 5.5. (a) The DTA curve or thermo gram is a plot between differential temperature and Time. (b) DTA curve may be endothermic (downward plot) or exothermic (upward plot).***

## Advantages

* It can be operated at very high temperature ranges.
* Highly sensitive technique
* Flexibility in crucible volume
* Both exothermic and endothermic reactions can be determined accurately.

## Disadvantages

* There is lot of uncertainity in transition reactions and heat of fusions upto 20 to 50%.
* Destructive limited range of samples time consuming usually not qualitative.

## Applications

* Used to identify the minerals both qualitatively and quantitatively.
* Rapid identification of the compositions of mixed clays.
* Polymers characteristics can be easily characterized.
* Degree of crystallinity can be measured.
* Degree of polymerization can be assessed.
* Many of the biological materials can be analyzed.
* Melting point, boiling point and temperatures of decomposition of organic compounds can be determined.
* Moreover, used to determine the thermal stability of inorganic compounds and complexes.

---

## Thermo-Mechanical Analysis

* A technique in which a deformation of the sample under non-oscillating stress is monitored against time or temperature while the temperature of the sample, in a specified atmosphere, is programmed.

* Thermo mechanical analysis easily and rapidly measures sample displacement as a function of temperature, time and applied force.

## Principle

* Thermo Mechanical Analysis (TMA) is used to measure the dimensional changes of a material as a function of temperature by applying stress.
* The stress may be compression, tension, flexure or torsion.

## Components

* Linear Variable Displacement Transducer (LVDT)
* Laser
* Optoelectronics, etc.
* probe (made up of quartz glass)
* Thermocouple Furnace
* Force generator

## Working

* The sample is inserted into the furnace and is touched by the probe which is connected with the Length Detector and the Force Generator.

* The construction of the push rod and sample holder depends on the mode of the measurements.

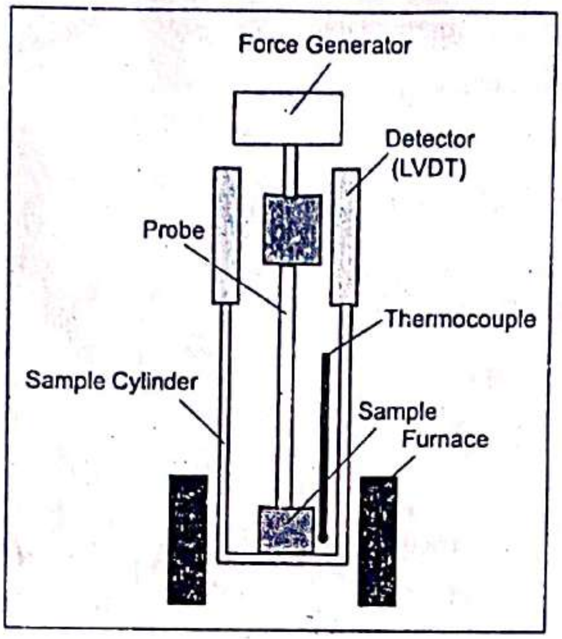



*Fig. 5.6. Working of Thermo mechanical analyser*

* The thermocouple for temperature measurement is located near the sample.
* The rate of 5°C/min is usually the maximum recommended value for good temperature equilibration across the specimen.
* The sample temperature is changed in the furnace by applying the force onto the sample from the Force Generator via Probe.
* The sample deformation such as Thermal Expansion and Softening with changing temperature is measured as the Probe displacement by the Length Detector.
* LVDT is used for Length Detection sensor.

* Every displacement of the push rod is transformed into an analog signal by the LVDT, converted to digital form and then recorded in the computer system, and finally presented by the software as a dimensional change versus time or temperature.

Advantages

* compact and light
* Low operating voltage
* Measures large deformation
* Large actuation force

Limitations

* Used only for solid samples
* creep occuring concurrently with normal dimensional changes
* Usage of prober probe
* Low operating speed

Applications

* Measurement of dimensional change
* co-efficient of linear thermal expansion
* Determination of material anisotropy
* Softening temperatures and glass transition
* Linear thermal expansion

# Dynamic Mechanical Analysis (DMA)

* Dynamic Mechanical Analysis is a technique where a small deformation is applied to a sample in a cyclic manner.
* This allows the materials response to stress, temperature, frequency and other values to be studied.
* Dynamic Mechanical Analysis (DMA) is an important technique used to measure the mechanical and viscoelastic properties of materials such as thermoplastics, thermosets, elastomers and metals.

## Principle

* A sinusoidal stress is applied and the strain in the material is measured, allowing one to determine the complex modulus.
* The temperature of the sample or the frequency of the stress are often varied leading to variations in the complex modulus; this approach can be used to locate the temperature of the material.

Types

* Forced resonance analyzers
* Free resonance analyzers

Mode

* Stress control
* Strain control

Components

* Linear variable Displacement Transducer
* Drive shaft or Probe
* Drive motor
* Stepper motor

Working

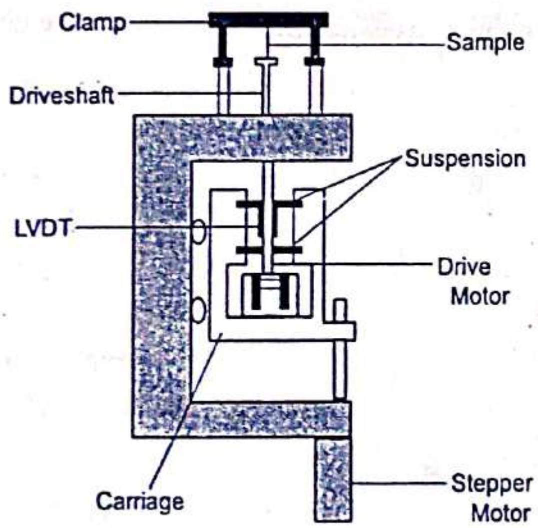



*Fig. 5.7. Cross section of Thermo mechanical dynamic analyser*

* The sample is clamped in the measurement head of the DMA instrument.

* During measurement, sinusoidal force is applied to the sample via the Probe or driving shaft.
* Deformation caused by the sinusoidal force is detected and the relation between the deformation and the applied force is measured.
* Properties such as elasticity and viscosity are calculated from the applied stress and strain plotted as a function of temperature or time.

Advantages

* It is an essential analytical technique to determine the viscoelastic properties of Polymers.
* Very soft and hard Samples are measured
* Allows accurate temperature measurement.
* It can Provide major and minor transitions of materials
* It is also more sensitive
* It is able to quickly scan and calculate the modulus for a range of temperatures.

Disadvantages

* It leads to calculation inaccuracy.
* The oscillating stress converts mechanical energy to heat and changes the temperature of the samples. ~~are slightly inaccurate.~~
* The final source of measurement uncertainity comes from computer error.

Applications

* Measurement of the glass transition temperature of polymers.
* varying the composition of monomers
* Mechanical properties in the relevant frequency range.
* Modulus information
* Measurement of different relaxations
* Molecular interaction
* Non linear properties
* Damping behaviour

## chemical Testing

* chemical testing provides a variety of Quantitative and qualitative services for Verification, identification and component analysis of ferrous and non-ferrous metals.

## Purpose of chemical testing

* chemical Trace Analysis
* Elemental Trace Analysis
* Failure Analysis
* contamination Analysis
* Materials Analysis and Testing
* Material verification
* Material Identification
* chemical composition Analysis

## X-ray Fluorescence

* X-ray fluorescence is a non-destructive analytical technique used to determine the elemental composition of materials.
* X-ray fluorescence analyzers determine the chemistry of a sample by measuring the fluorescent X-ray emitted from a sample when it is excited by a primary X-ray source.

* The phenomenon is widely used for elemental analysis and chemical analysis, particularly in the investigation of metals, glass, ceramics and building materials and for research in geochemistry.

## Principle

* X-ray fluorescence (XRF) is the emission of characteristic "secondary" X-rays from a material that has been excited by being bombarded with high-energy X-rays or gamma rays.

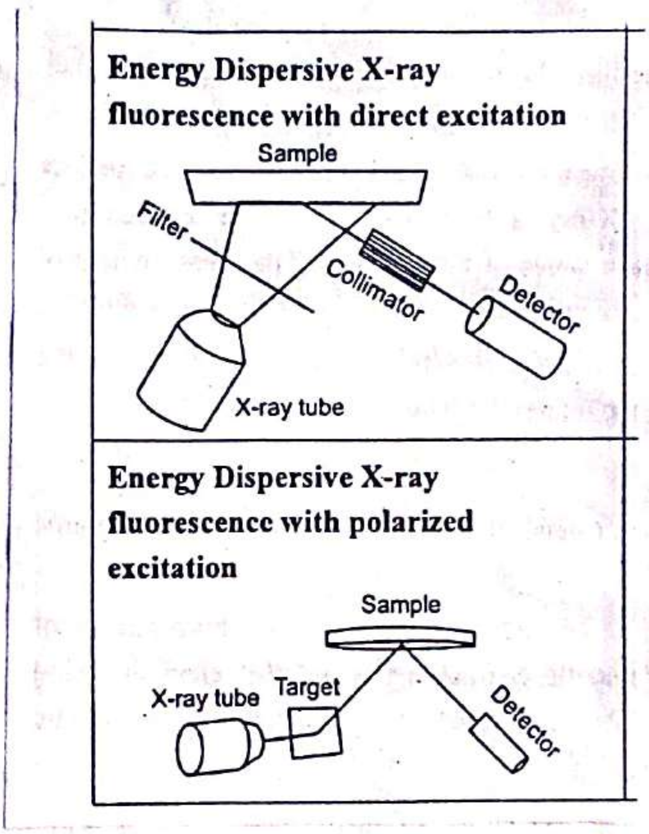
## Components

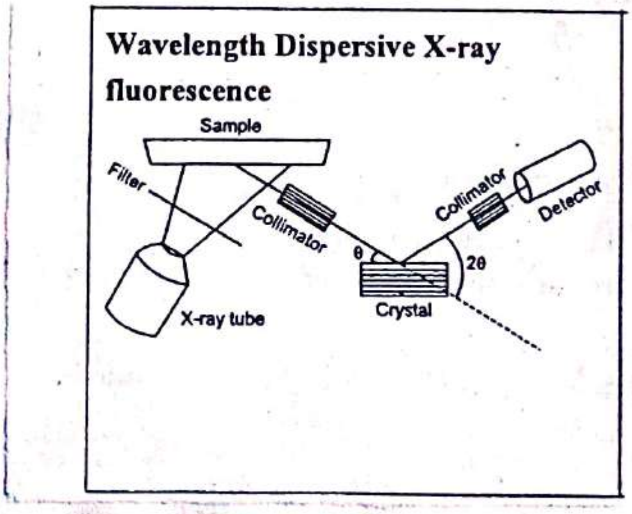
* source of X-rays used to irradiate the sample.
* Wave lengths are typically in the range 0.01 to 10 nm, which is equivalent to energies of 125 KeV to 0.125 KeV.
* Detection equipped by Gas-filled detectors, semiconductor detector, Scintillation detector, a photographic plate.

## Types of XRF spectroscopy

The XRF spectroscopy differs primarily by detection and analyzing.

* Energy Dispersive XRF
* Wavelength Dispersive XRF



## Energy Dispersive X-ray fluorescence with direct excitation

* An energy dispersive de-tection system directly measures the different energies of the emitted X-rays from the sample.
* By counting and plotting the relative numbers of X-rays at each energy an XRF spectrum is generated.

## Energy Dispersive X-ray fluorescence (XRF) with Polarized excitation

* The detector must be perpendicular to the plane determined by the tube, target and sample.
* The most important effect is that by deflecting the X-ray radiation by 90°, the radiation is Polarized and the spectral background in the spectrum is reduced.

## Wave length Dispersive X-ray fluorescence (XRF)

* The X-rays are directed to a crystal, which diffracts the X-rays in different directions according to their wavelengths.

* On a sequential system a detector is placed at a fixed position, and the crystal is rotated so that different wavelengths are picked up by the detector.

Working

* A solid or a liquid sample is irradiated with high-energy X-rays from a controlled X-ray tube.
* When an atom in the sample is struck with an X-ray of sufficient energy, an electron from one of the atom's inner orbital shells is removed.
* The atom regains stability, filling the vacancy left in the inner orbital shell with an electron from one of the atom's higher energy orbital shells.
* The electron drops to the lower energy state by releasing a fluorescent X-ray.
* The energy of this X-ray is equal to the specific difference in energy between two quantum states of the electron.
* The measurement of this energy is the basis of XRF analysis.

* The intensity of each Characteristic radiation is directly related to the amount of each element in the material.

Advantages

* Simple spectra analysis
* XRF is a Versatile and rapid technique
* It is non destructive method of chemical analysis
* It is precise and with skilled operations it is accurate.
* Applicable to a wide variety of Samples from Powders to liquids.
* It is convenient and economical to use

Disadvantages

* It fairly high limits of detection when Compared to other methods.
* XRF analyzes cannot distinguish Variations among isotopes of an element.
* XRF analyzes Cannot distinguish ions of the Same element in different Valence states.
* Instrumentation is fairly expensive.

## Applications

* It is a method of elemental analysis with atomic number greater than 12.
* Quantitative analysis can be carried out by measuring the intensity of fluoresance at the wavelength characteristics of the element being determined.
* Research in igneous, sedimentary and metamorphic petrology, soil surveys, mining, cement production, ceramic and glass manufacturing.
* Metallurgy
* Environmental studies
* Petroleum industry
* Field analysis in geological and environmental studies.
* Bulk chemical analyses of major elements and trace elements.

## Elemental analysis by inductively coupled Plasma

### Elemental Analysis

* Elemental analysis is a process where a sample of some material is analyzed for its elemental and sometimes isotopic composition.
* Elemental analysis can be qualitative and it can be quantitative.
* Elemental analysis falls within the ambit of analytical chemistry, the set of instruments involved in deciphering the chemical nature of our world.

### Plasma

* The excitation source must desolvate, atomize and excite the analyte atoms.
* A variety of excitation sources are flame, arc and plasma.

### Inductively-coupled plasma

* An inductively coupled plasma (ICP) or transformer coupled plasma (TCP) is a type of plasma source in which the energy is supplied by electric currents which are produced by electromagnetic induction, that is by time-varying magnetic fields.

* The most commonly used ion source for plasma spectrometry, the ICP, is produced by flowing an inert gas, typically argon, through a water cooled induction coil which has a high-frequency field (27 MHz) running through it.
* An inductively coupled plasma (ICP) is a very high temperature (7000 – 8000 K). excitation source.
* ICP sources are used to excite atoms for atomic-emission spectroscopy and to ionize atoms for mass spectrometry.

Production of Plasma

* Inductively coupled discharge also uses RF power supply like capacitively coupled discharge.

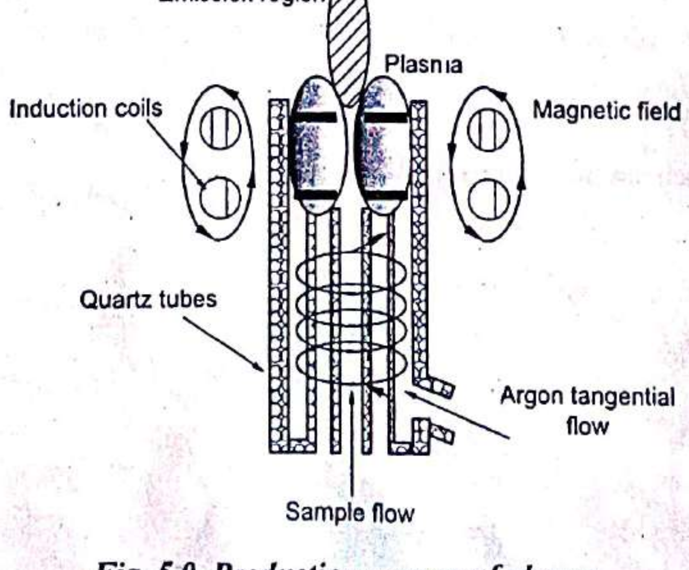



*Fig. 5.9. Production process of plasma*

* A radio frequency (RF) generator (typically 1-5 KW @ 27 MHz) produces an oscillating current in an induction coil that wraps around the tubes.
* For a commonly used cylindrical plasma chamber shown below, antenna is usually wrapped around the electrically insulating chamber wall.
* RF generator drives high alternating current through coil antenna, which creates an alternating magnetic field within the plasma chamber.
* Oscillating magnetic field will generate an oscillating electric field in the plasma chamber.
* Eventually, the electric field will accelerate the electrons and generate Plasma.
* The magnetic field in turn sets up an oscillating current in the ions and electrons of the support gas (argon).
* As the ions and electrons collide with other atoms in the support gas.
* Since the excitation force is delivered through magnetic field, inductively coupled discharge is also called "H-discharge".

## characteristics of optically coupled Plasma 

* High temperature (7000 - 8000 K)
* High electron density (1014-1016 cm)
* Appreciable degree of ionization for many elements
* Simultaneous multielement capability
* Low background emission and relatively low chemical interference
* High Stability leading to excellent accuracy and Precision
* Excellent detection limits for most elements (0.1 - 100 ng mL-1)
* wide linear dynamic range (LDR)
* Applicable to the refractory elements cost-effective analyzes

## optical Emission spectroscopy

* optical Emission spectroscopy or OES analysis, is a rapid method for determining the elemental composition of a variety of metals and alloys.

Based on excitation source optical Emission Spectroscopy is classified as,

* Inductively coupled optical Emission Spectroscopy

* Glow Discharge optical Emission spectroscopy or Glow Discharge MS (GD-MS)

* Arc spark optical Emission Spectroscopy

* Flame emission spectroscopy

Inductively coupled Plasma optical emission Spectroscopy

* The Inductively coupled plasma optical Emission spectroscopy (ICP-OES) analysis method uses high-frequency inductively coupled Plasma as the light source, and is ideal for the element analysis of Sample Solutions.

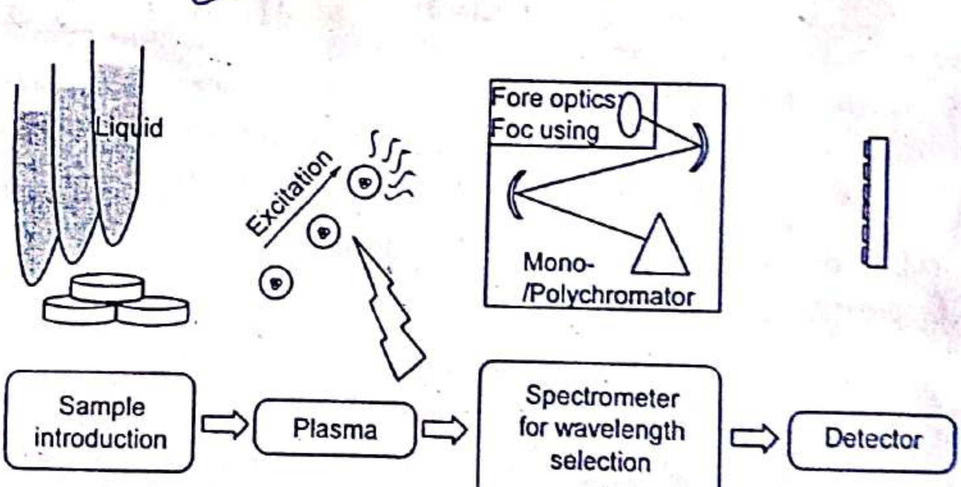



*Fig. 5.10. Flow diagram of ICP-OES*

Principle:-

* when plasma energy is given to an analysis sample from outside, the component elements is excited.

* When the excited atoms return to low energy position, emission rays are released and the emission rays that correspond to the photon wavelength are measured.

Components

* Nebulizer
* Pump
* Spray chambers
* Drains

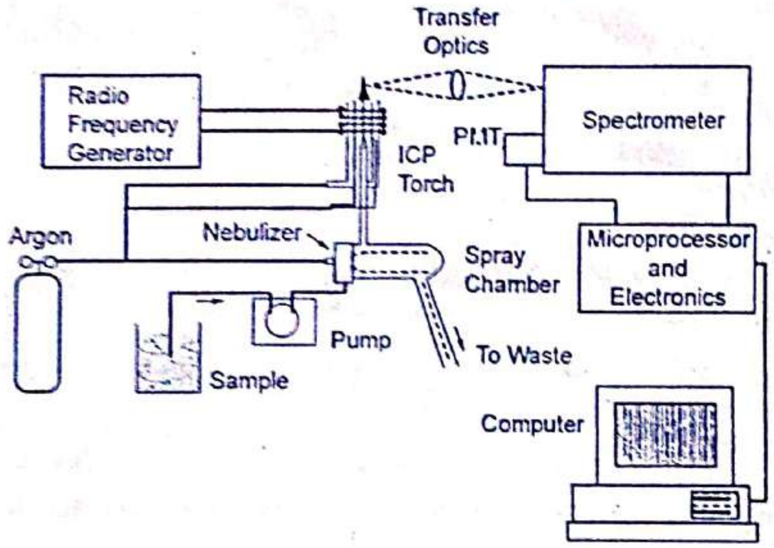



*Fig. 5.11. A representation of the layout of a typical ICP-OES instrument.*

Working of ICP-OES

* The first step in an analysis is to prepare the samples and standards for introduction to the ICP.
* This step depends on the physical and chemical characteristics of the samples.

* The next step in the analysis concerns the sample introduction method and hardware to be used.
* For most ICP-OES analyses, the standard sample introduction system provided with the instrument will be sufficient.
* In inductively coupled plasma-optical emission spectrometry, the sample is usually transported into the instrument as a stream of liquid sample.
* Inside the instrument, the liquid is converted into an aerosol through a process known as nebulization.
* The sample aerosol is then transported to the plasma where it is desolvated, vaporized, atomized and excited or ionized by the plasma.
* The excited atoms and ions emit their characteristic radiation which is collected by a device that sorts the radiation by wavelength.

* The radiation is detected and turned into electronic signals that are converted into concentration information for the analyst.
* The next step in the development of an analysis methodology is to program the instrument, using the computer software provided with the instrument, to perform the data collection and processing steps.
* To do this, decisions must be made concerning the operating conditions, wavelength selection, instrument calibration, emission measurement and the actual sample analysis.

Advantages

* Extremely high sensitivity.
* Almost full elemental coverage without need for specific excitation sources.
* Linear range of several orders of magnitude
* very accurate quantification at low concentrations.
* Simultaneous determination of multiple elements in each sample.

* Complementary analysis to techniques like XRF
* Large dynamic linear range
* Low chemical and matrix interference effects

Disadvantages

* Initial progress is often time consuming
* In the case of failure analyses method development will often be necessary each time a new sample type is encountered.
* Relatively long analysis times
* The method is inherently destructive

Applications

* Trace analysis of environmental soil and water samples.
* Assessment of metal ores for mass balances and process control
* Boron and Lithia in glasses
* Forensic analysis
* Trace analysis of food and drink samples such as: metals in wine; and elements bound to proteins.
* Determination of contaminants in high-purity Al

# Inductively coupled Plasma mass Spectrometry 

* Inductively coupled Plasma mass Spectrometry (ICP-MS) is an instrumental analytical technique based on the use of a high temperature ionisation source (ICP) coupled to a mass spectrometer.
* It is an elemental analysis technology capable of detecting most of the periodic table of elements at milligram to Nano gram levels per liter.
* It is used in a Variety of industries including, but not limited to environmental monitoring, geochemical analysis, metallurgy, Pharmaceutical analysis and clinical research.

## Principle

* It is a type of mass spectrometry that uses an inductively coupled plasma to ionize the sample.
* It atomizes the sample and creates atomic and small Polyatomic ions, which are then detected.

* It is known and used for its ability to detect metals and several non-metals in liquid samples at very low concentrations.
* It can detect different isotopes of the same element, which makes it a versatile tool in isotopic labeling.

Components

* Peristaltic pump
* Nebulizer and spray chamber
* Torch
* Plasma Ionization source
* Interface Region
* Ion Focusing Region
* Mass Analyzer
* Spectral Interferences
* collision Reaction cell
* Ion Detectors

Working

* The sample solution is introduced into the device by means of a peristaltic pump.
* There it becomes nebulized in a spray chamber.

* The resulting aerosol is injected into an argon-Plasma that has a temperature of 6000 - 8000 K.
* Inside the Plasma torch, solution is removed from the Sample and also atomization and ionization occur.

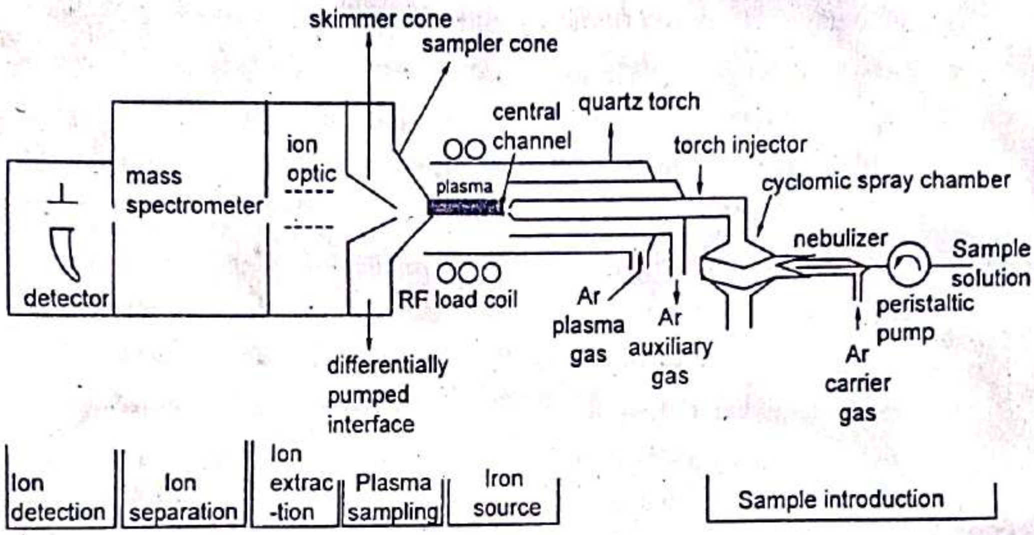



*Fig. 5.12. Working flow of ICP-MS*

* To be Processed efficiently in the Plasma, Samples must be in either gas or vapor form.
* Only a small amount part of the ions produced in the Plasma further penetrate to the mass - spectrometer part.
* After mass separation, ions must be detected and amplified in order to determine their intensities.

* Electron multipliers can detect extremely small ion currents, including even single ions, coming from the mass analyzer.
* They operate on the principle of secondary electron emission, in which charged particles with sufficient energy incident on a 'dynode' stimulate the emission of electrons from the surface.

Advantages

* Quantitative analysis is the fundamental tool used to determine analyte concentrations in unknown samples.
* Increased sensitivity and wide dynamic range
* Extremely low detection limits
* A large linear range
* Possibilities to detect isotope composition of elements
* Wide Elemental coverage
* Extremely Low Detection Limits. (mg/L)
* Fast Analysis times
* Simple spectra
* Isotopic information
* High Productivity

## Disadvantages

* High capital cost of the instrumentation
* Lower precision compared with atomic absorption spectrometry
* Total dissolved salts should be less than 1000 ppm
* Severe matrix effects
* Heavier elements such as lead are well-suited for ICP-MS analysis, whereas lighter elements are prone to more interference.

## Applications

* simple metal analysis during metal based drug development
* Impurity limit tests
* Metals present in Active Pharmaceutical Ingredients
* Quality control Tests of natural products for toxic impurities testing
* Monitoring metabolites of an administered drug.

* Detection of metal impurities from leachable packaging material
* For elemental Speciation
* Pharmaceutical waste water monitoring